

OUTLINES OF ANALYTICAL METHODS

A guide to the occurrence, significance,
sampling and analysis of chemical and
microbiological parameters in water,
sediment, soil, vegetation and air.

June, 1981



Ontario

Ministry
of the
Environment

The Honourable
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Minister

Graham W. S. Scott, Q.C.,
Deputy Minister

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**COORDINATED BY
WATER QUALITY SECTION**

Ontario Ministry of the Environment

Laboratory Services Branch

June, 1981.



FOREWORD

Information on analytical methods is often required in order to correctly interpret data and to prepare reports. This collection of analytical outlines has been prepared in order to provide information in a brief, non-technical form and to assist the sampler in understanding the meanings behind certain chemical and microbiological parameters as well as their limitations.

Methods in this manual are suitable for routine chemical and microbiological analyses on several sample matrices and include specific information as to proper sampling and preservation techniques. Several non-routine tests, not listed in this manual, can be performed by laboratory staff upon special request. Information about these tests may be obtained from the Laboratory Services Branch, Ontario Ministry of the Environment, Box 213, Rexdale, Ontario, M9W 5L1.

Individual test descriptions are revised periodically as needed. Samplers are urged to fill out the form at the back of this manual and return it so that they can be notified of any revisions.

ACKNOWLEDGEMENTS

I wish to acknowledge the following people who contributed a great deal of time and effort towards the production of these outlines. Michael Young contributed the microbiology outlines. Dr. Barry Loescher and Mrs. Pat Lachmaniuk aided in the Inorganic Trace Contaminants outlines, Dr. Ed Adamek and Mr. Bob Gutteridge helped with the Organic Trace Contaminants outlines, Mrs. Joan Crowther helped with the Water Quality Section outlines and Pat Baulu and Joe Osborne helped with the Pesticides outlines. Ms. Anne Bohdanowicz contributed much of the data as well as a number of the test write-ups. A special acknowledgement should be made to Mrs. Fran Hall who did all the typing.

I would also like to acknowledge the contributors to the preceding manuals who put a great deal of effort into the compilation of the format and content.

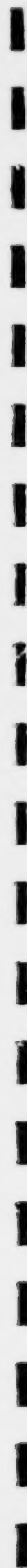
W. B. Moody
Laboratory Services Branch

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INTRODUCTION

The various laboratories of the Laboratory Services Branch are equipped to perform a large number of standard chemical and microbiological analyses on domestic water supplies, surface waters, ground waters, domestic and industrial wastes as well as air, soil, sediment, vegetation and sludge samples. Special analyses required for research studies and unusual pollution problems may also be provided upon consultation with laboratory staff.

Three regional laboratories at Kingston, London and Thunder Bay are operational in addition to the main Toronto Laboratory. The boundaries of the districts serviced by these laboratories are shown in Figure 1. Samples collected in Regions 2, 3 and 5 are analyzed in Toronto. Samples collected in Regions 1, 4 or 6 are analyzed in London, Kingston and Thunder Bay respectively when the analytical capability is available. If such capability is not available, these samples are analyzed in Toronto.

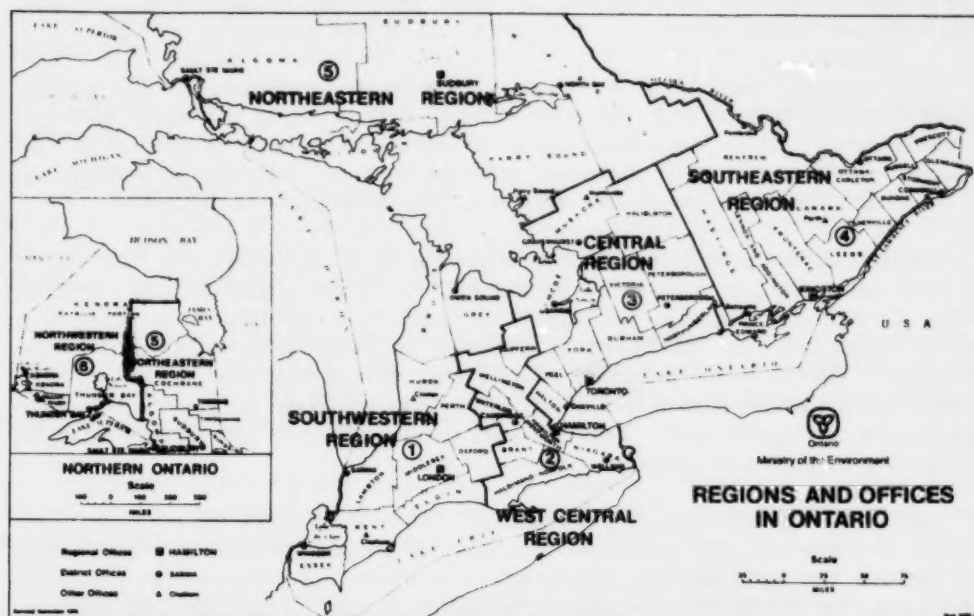


Figure 1

The tests performed by each regional laboratory as well as the sectional laboratories in Toronto (W = Water Quality; ITC = Inorganic Trace Contaminants; OTC = Organic Trace Contaminants; P = Pesticides; and M = Microbiology) are outlined in Table 1. Field samplers should ensure that a laboratory is capable of analyzing the parameter(s) requested before shipping. Mobile laboratories or field stations, such as at Sudbury or Dorset, may be provided to perform a limited number of tests in conjunction with major surveys or studies.

CHEMICAL TESTING

Collection and Perishability of Samples

The method of collecting the sample in the field is the concern of the individual in charge of the particular survey. If the sample is not representative of the whole, the subsequent analyses will be worthless. Care must be taken to prevent contamination of the sample by sampling devices, motor exhausts, disturbing the sediment beds of lakes etc. Furthermore, it must be realized that the composition of most samples will change with time; thus the sample must be quickly forwarded to the laboratory for analyses. The perishability of a sample may be due to biological activity, chemical precipitation, leaching of contamination from containers, adsorption on the walls of containers, chemical reactions between constituents and volatility. Tests such as dissolved oxygen, dissolved carbon dioxide, free chlorine and pH are examples of extremely perishable parameters. Field analyses for these parameters are recommended whenever possible.

For some parameters, the use of a preservative or refrigeration is recommended. These techniques may delay perishability somewhat, but the sample should still be transported to the laboratory as quickly as possible.

For samples which do not have a preservative already in the bottle, rinsing both the bottle and cap with sample two or three times is strongly recommended. This procedure helps to equilibrate the sample with the container walls and hence "container effects" such as leaching or adsorption can be minimized. This procedure may also help reduce contamination.

In view of the probable chemical degradation of a sample, the laboratory analyzes each sample as rapidly as present facilities permit. The average number of analyses possible per day is included in each method outline; this number could only be increased by reassigning staff from other analytical functions. This number provides a guideline which will allow samplers to regulate their sample inputs and avoid pre-empting a large portion of the daily capacity for any given test. Your co-operation will assist the laboratories in maintaining a quick and efficient analytical service.

Sampling information is provided in Table 2 as well as each method outline. If there are any questions, the sampler should consult with laboratory staff before the samples are collected. For more information on sampling techniques, the reader is referred to "A Guide to the Collection and Submission of Samples for Laboratory Analysis" published by the Ontario Ministry of the Environment, Laboratory Services Branch.

Sample Containers and Preservation Techniques

In general, water samples are collected in 1 liter glass bottles, but some analyses require plastic containers. Many procedures call for sample preservation. Table 2 includes a summary of both the preferred container type as well as the preservation techniques for each parameter. The individual outlines also provide this information.

While some parameters are stabilized by a certain preservation technique others may be destroyed or altered.

It is the responsibility of the sampler to obtain the proper sample container and to perform the proper preservation technique.

Sludge samples should be collected in wide mouth glass jars and never filled more than half way. The extra space is required as an expansion zone for gaseous products that may be formed. Failure to submit samples in this manner may result in container explosion in transit or at the laboratory. **Overfilled sludge samples are discarded without analysis.** Sludge samples for trace organic analyses other than BOD, COD and volatile acids must have a foil lined cap.

Glass containers are required for domestic waste and industrial waste samples. **For the protection of Laboratory Staff, it is imperative that information about hazardous substances in the sample be clearly written on the sample container.** A proper label should be placed on the bottle (toxic, explosive, corrosive, etc).

Special acid washed plastic containers are required for trace level heavy metal analyses. For domestic water, surface water and precipitation samples, heavy metals with the exception of mercury, are preserved by adding 20 drops (1 mL) of concentrated nitric acid to one liter of sample after the sample is in the bottle. **Never add acid to an empty plastic container prior to sampling** as strong acid will activate plastic surfaces in such a way that some heavy metals may be adsorbed onto the container walls. The use of foil lined caps should be avoided when sampling for trace quantities of heavy metals.

Standard glass bottles with non-metal liners may be used for all other routine samples requiring metal analyses.

Precipitation sampling should be conducted using sample collectors designed to catch precipitation as it falls. Once the sample has been collected, it is transferred to polystyrene bottles. Special acid washed plastic containers are used for trace level metal analyses.

Aqueous samples for organic analyses should be submitted in 1 liter glass bottles with foil lined caps. The sample should have no head space and should be refrigerated but not frozen. If analysis of hydrocarbon gases is required, approximately 20% head space should be provided.

PCB and pesticide samples must be collected in new 1 L brown glass bottles which have been previously rinsed with methylene chloride and then acetone. These must have foil lined caps.

Asbestos samples should be collected in new 1 L plastic bottles.

A clean, sealed polyethylene dustfall collector jar should be used to collect dustfall. In summer 1 mg/L of CuSO_4 may be added as an algal or fungal inhibitor provided it will not affect the desired analysis. Since the container must be kept in an upright position, shipping via CN or CP is not feasible.

Hi-Vol filters should be removed from their cassettes, folded in half along the 20 cm width, particulate side inwards and placed in a kraft paper envelope. The filter envelope must contain the following information: Station number, Hi-Vol instrument number, date and time of exposure, filter number, operator, flow readings at start-up and shut-down; and any comments peculiar to the sampling period.

Fluoridation rates are measured using the appropriate candle while sulphation rates are measured using the appropriate plate. After exposure the candle or plate should be replaced in its protective container and returned to the laboratory.

Vegetation samples should be placed in perforated polyethylene bags and transferred to refrigerated storage as quickly as possible.

Any clean glass or plastic container is acceptable for sediment, soil or biomaterial samples. The pomade jar and twirl-top bags are the most commonly used. For short term preservation, refrigeration at 4 °C is recommended. Drying, as a preservation technique is recommended except for samples requiring analysis of potentially volatile parameters. If drying in the field is not practical, the laboratory should be notified so that the samples can be dried without delay.

In many instances, generally arising from citizens' complaints, it becomes necessary for field personnel to collect samples for constituent identification by means of microscopic, X-ray diffraction, electron probe and other techniques. The types of material normally encountered are visible solids present in air or water, that are a cause of nuisance or concern to the complainant. To facilitate the collection of these samples, a sampling kit is available from the Laboratory Services Branch for each district office in the province. Supervisory personnel should obtain and maintain this kit. Detailed sampling instructions are included in the kit.

A general summary of sampling containers available from Central Stores is listed in **Table 3**.

Sample Volumes

Each method outline specifies the volume of sample required for proper analysis on all sample types. The volumes are also listed in Table 2. For a sample containing low concentrations of a substance, the largest practical volume is tested in order to provide a sufficient quantity of that substance for reliable detection.

The sampler is expected to calculate the total volume of sample required by summing the individual volumes (from Table 2) for all the analyses requested and to submit the appropriate quantity. A number of tests may be determined simultaneously on the same aliquot.

1. Both total phosphorus and total kjeldahl nitrogen can be determined on a single 75 mL aliquot.
2. Nitrate-, nitrite- and ammonia-nitrogen and filtered reactive phosphorus can all be determined on a single 75 mL aliquot.
3. Both sodium and potassium (or calcium and magnesium) can be determined on a single 50 (or 100) mL aliquot.
4. Aluminum, copper, cadmium, cobalt, chromium, iron, lithium, molybdenum, manganese, nickel, silver, strontium, zinc, tin, barium, beryllium, gold, lead, thallium, vanadium and titanium, can all be analyzed on a single 500 mL aliquot if necessary.
5. Dissolved organic and dissolved inorganic carbon can be determined on the same 50 mL aliquot.

Samplers are asked to submit at least 180 mL of sample in excess of their original total estimate to allow for rinsing of tubes and possible repeat analyses. Failure to submit sufficient sample volume will result in "Sample Exhausted" being marked on analysis report sheets. The sampler must also use the proper containers.

In certain cases, where the sampler is unable to obtain sufficient sample volume or when resampling is impossible, analysis may still be performed if special techniques are used in the laboratory. This can only be achieved after consultation with laboratory staff before sample submission.

Sample Submission

Sample Bottles - All sample bottles must be clearly labelled and contain the following information:

1. A sample (sender's) number.
2. Some other identification, normally the sample source or type (e.g. "Lake Temagami - Sharp Rock Inlet").
3. Presence of any chemical preservative added; all others will be kept refrigerated or frozen until time of analysis.
4. When appropriate, indication of the specific analyses to be performed on that sample bottle (e.g. "Nutrients", "Phenolics").

5. Proper cautionary notes to analysts, if hazardous substances are present (cyanide, strong acids or bases, PCB's etc.).

Submission Forms - Submission and Request for Analysis forms MUST accompany all samples and must include the following information:

Submission Form-

- the first and last field sample numbers
- the sample program code
- the laboratory to which the samples are sent
- the priority of the submission, high or normal
- the sampling agency code
- the date the submission was sent to the laboratory (Day/Month/Year)
- the name of the sampler
- the phone number of the sampler
- the client code of the person or group responsible for the submission or the name, address, phone number and establishment of the person to whom the report should be sent
- the client codes or names and addresses of other people to whom reports should be sent

Request for Analysis Form-

- the field sample number
- the sample type code
- the number of containers sent
- the sampling date (day/month/year) and time
- the sample location or station
- a sample description such as soil, sewage, etc.
- the test group(s) requested
- any deletions from the test group, on the same line as the test group
- any individual test additions

If a group of related samples is submitted, a map of the area or the relative location of waste inputs is very helpful to the laboratory staff. Please number the samples in a logical order, e.g. downstream in a river. All analyses are routinely screened for anomalous results before they are released. Suspicious results might be improperly rejected whereas some details as to the location would confirm their validity.

When a sample is sent to the laboratory a description of known constituents should be included on the submission sheet, particularly an unusual one such as an industrial waste contaminant. Interferences (reactions which produce false analytical results) can be eliminated by pretreatment if the analyst is forewarned. Samples known to contain cyanide, arsenic, PCB's or other toxic materials should be clearly marked for the protection of laboratory personnel.

Identification of unknown contaminants is very time consuming. Samples should be as large as possible to allow a wide range of exploratory tests. The sampler should indicate whether qualitative or quantitative results are required. Any available information concerning the sampling point, possible contaminants, and industries implicated is extremely important for such samples.

Samples which are not homogeneous present analytical difficulties because it is virtually impossible to take a representative aliquot. If the sampler is only interested in one phase (aqueous, solid or immiscible organic), he should label the submission form accordingly. Otherwise the laboratory will consider the whole sample, and take aliquots of the mixture.

Erroneous results for trace heavy metals in water will be obtained if particulate material is inadvertently present in the sample, e.g. for samples of groundwater in landfill monitoring wells. Great care must be taken to minimize such contamination. Field filtration may be necessary prior to addition of acid preservative.

MICROBIOLOGICAL TESTING

Collection of Samples

Sterile sampling bottles are available from Central Stores in Toronto. For special studies, alternate bottles are obtainable directly from the Microbiology Section. It is the responsibility of the sampler to use aseptic techniques when handling these sterile bottles. Inaccurate results are obtained when contamination occurs through faulty handling of sterile containers.

Surface Samples

Clamp the bottle onto a sampling pole before removing the cap. Touch only the outer surface of the cap when opening the bottle. The inner lip and liner must not come in contact with anything except the atmosphere. If the inner surfaces of the cap or bottle lip are accidentally touched, the sample has been contaminated and should not be submitted. It is a recommended procedure to hold the cap with the fingertips until the sample has been taken. The cap must not be set down while the sample is being taken as this will result in contamination.

Surface sampling is accomplished by quickly lowering the sample bottle below the surface of the water and holding it there until all bubbles have stopped coming to the surface. When sampling a stream or river, place the bottle with the mouth facing into the current. When sampling near shore, care should be taken to get a sample which has not been contaminated with sediment. The bottle is then removed from the water, the level adjusted to the top of the label, immediately capped, and then unclamped from the sampling pole. Samples must be collected using this prescribed technique. The use of a dipper or other sampling device will result in contamination.

Depth Samples

Depth samples are taken using sterile sampling bulbs which can be obtained from the Microbiology Section in Toronto. Bulbs should be used as quickly as possible. If not used, they should be returned to the Microbiology Section within a maximum of two weeks, otherwise the rubber will crack and the bulb will not open. The same care that is used with sampling bottles must be used in the handling of bulbs. The glass plugs supplied have been sterilized within a cellophane envelope and must not come in contact with any contaminated surfaces when they are being removed from the cellophane envelope. If, for some reason, the sampler should run out of glass rods, he may dip the metal plug into alcohol and flame it. After flaming, the plug is immediately inserted into the bulb, taking the usual precautions when handling sterile equipment. The use of the metal plug is discouraged and it should only be used in rare instances when the sample could not possibly be obtained at a later time in the correct manner.

Tap Samples

Prior to sampling from a tap, the water should be allowed to run at full flow for approximately two minutes. The strong flow will clean out residual contamination around the orifice of the tap thus providing for a more representative sample. The water pressure may then be reduced to permit

taking the sample without excessive splashing which could result in contamination of the sample.

Fill the bottle to the top of the label, being certain that the mouth of the bottle does not come in contact with the tap or any contaminated surface. The cap must also be handled aseptically as described previously.

Perishability

While being transported to the laboratory, samples undergo changes (increases or decreases) in bacterial population, depending upon the initial bacterial density, the presence of growth-promoting or toxic substances in the water and the environmental conditions under which the samples are held. Analyses should be performed as soon as possible after collection, preferably within six hours, but not exceeding 24 hours.

Drinking water, waste and river samples that require transportation by public carriers received 24 to 72 hours after collection can be analyzed. Samples received after three days are rejected. Negative coliform results for drinking water samples arriving more than 48 hours after collection should be considered incorrect and fresh samples should be collected.

Sample Containers and Preservation Techniques

Presterilized 180 ml bottles with red labels usually provide adequate volume for the routine analysis of water from distribution systems, surface waters and well waters. It is particularly important that chlorinated waters, or waters in which a chlorine residual is suspected, be sampled in bottles containing sodium thiosulphate as a preservative (red label). Sodium thiosulphate will neutralize the disinfecting capability of the chlorine thereby preserving the existing microbial population at the time of sampling. Before samples are collected for sulphur oxidizing bacterial analyses, the Microbiology Section should be contacted to determine whether thiosulphate should be used or not.

All samples should be collected early in the week and shipped to the laboratory without delay. During spring, summer and fall, samples must be packed in ice to minimize biological activity. During winter, samples should be packed in an insulated container that will prevent freezing while still keeping them cold.

Sample Volumes

In general, one bottle or bulb per sample provides sufficient volume for standard analysis. If, however, the bacterial levels expected are very low or extra parameters are being requested, then additional samples may be required. Consultation with the Microbiology Section is advisable in such cases. When a duplicate sample is being taken, it should be obtained at the same time as the first sample. This can be achieved for surface samples by clamping two bottles on a sampling pole and for depth samples by placing two bulb samplers on the sampling line in a 'piggyback' fashion.

For sample submission of microbiology samples, refer to the section on sample submission.

SHIPPING PROCEDURES

CN Express and CP Express provide the fastest and most reliable service for the shipment of environmental water samples in Ontario. Air Express, parcel post, bus companies and other services discourage the shipment of water samples because of the damage caused to other shipments when breakage occurs. For samples which must be kept in an upright position, shipping by CN, CP, etc., is not feasible.

Contract numbers are important as they provide the only means for tracing a lost shipment. Every shipment is assigned a contract number at the Express Depot, but it is generally up to the sampler to attach this contract number to each carton of his shipment. Identification stickers are provided by the Express companies upon request. Samplers are urged to keep a record of all their contract numbers.

The following addresses should be used when shipping samples to the various laboratories.

1. Central Region - Main Toronto Laboratory

Ontario Ministry of the Environment,
Central Stores,
Resources Road,
Highway 401 and Islington,
Rexdale, Ontario.

2. Southwestern Region - London Laboratory

Ontario Ministry of the Environment,
Southwestern Regional Laboratory,
985 Adelaide St. S.
London, Ontario.

3. Northwestern Region - Thunder Bay Laboratory

Ontario Ministry of the Environment,
Thunder Bay Regional Laboratory,
421 James St. South
Thunder Bay, "F", Ontario.

4. Southeastern Region - Kingston Laboratory

Ontario Ministry of the Environment,
Southeastern Regional Laboratory,
133 Dalton St.,
Kingston, Ontario.

Sample Enquires

All samples received by the Laboratory Branch are assigned numerical codes according to sample type. When the analyses are completed, the results are entered onto the report sheets, checked by the scientist or technician in charge, and sent to the people listed on the sample submission form. All original submission sheets are retained in the sample reception files. Laboratory staff are prepared to answer questions regarding the receipt and progress of samples but require the following information:

- the submission number if known.
- the name or numerical code of the sampling program.
- the date the sample was submitted to the laboratory.
- the municipality from which the sample was taken
- the sampler's name.
- sample identification number including laboratory number if known.
- the sample type (water, sewage, Great Lakes, etc.).
- the tests requested

The following is a listing of telephone numbers where enquiries are handled most frequently:

Water Quality Section

416-248-3512

Sewage and Sludge	Ben Cheung Peter Millar	
Well and Drinking Water	Stuart Barnes	
Rivers and Lakes	Stella Tracy Stan Wisz	
Precipitation	Jenifer McBride	
Electron Microscopy Liquid Scintillation	Paul Roberts	
Legal Samples	Mike Rawlings W.B. Moody Frank Tomassini	
Dorset Laboratory	Charlie Chun	705-766-2213
Sudbury Laboratory	Michelle Brideau	705-675-4501

Inorganic Trace Contaminants Section

Legal Samples Priority Samples	Jim Pimenta	416-248-7101
Hazardous Materials, Industrial Wastes	Moselhy M. Moselhy	416-248-7101
Sediments, Landfill, Solid Wastes, Sewage, Soils, Leach Tests, Aquatic Vegetation	Frank Darcel	416-248-3346
Metals Coordinator	Barry Loescher	416-248-3775

Precipitation Samples

Mercury (All samples) Fish, Biomaterials	Darryl Russell	416-248-3023
Hi-Vol, Dustfall, Plates Legal Samples (Air)	Brian Foster	416-248-3346
Vegetation and Soils (Air Fallout)	Ron Wills	416-248-3346
Complaint Samples Identification - Air, Water	George Reese	416-248-3775
Emission Spectrometer Spectrographic Scans ICP Atomic Emission	Dave Boomer	416-248-3029
Water Samples - Status	Cliff Lee	416-248-3775

Organic Trace Contaminants

Oil and Petroleum Hydro- carbons, Organic Char- acterisation Court Cases	George Wyhovszky	416-248-3469
Haloforms in Water, PAH Air Sampling	Ed Adamek Bob Gutteridge	416-248-3204
Status of Samples through Lab.	George Wyhovszky	416-248-3469

Pesticides Section

Organochlorines	George Crawford	416-248-3743
Carbamates, Phenyl Urea	Pat Baulu	416-248-3614
Triazines, Organophosphates Chlorophenxy Acids		
PCB, PBB, HCB, Chlorinated Aromatics	Joe Osborne	416-248-3743

Microbiology

416-248-3008

Water Lab.	S. Debrececi
Biohazards Lab.	D. Rokosh
Virology Lab.	G. Jenkins
Rivers and Wastes	G. Tanai
Legal Samples	J. Clark

Lakes and Surveys	S. Janhurst	
Taxonomy, Acid Rain	M. Young	
Nuisance Organisms Complaint Samples	G. Horsnell	
Dorset Laboratory	D. Evans	705-766-2712
Regional Laboratories		
Kingston		613-549-4000
Chief Chemistry Microbiology	Stan MacBeth Dave Ferguson Art Ley	
London		519-681-3600
Chief Chemistry Microbiology	Dave Glutek Walter Cook Gary Palmateer	
Thunder Bay		807-475-1275
Chief Chemistry Microbiology	Albert Perras Stu Irwin	

ANALYTICAL OUTLINES

The chemical tests performed by the various laboratories are described in the following outlines which are listed alphabetically according to the assigned test titles on Tables 1 and 2. Each outline contains a variety of information which can be used by samplers to choose the most applicable parameters for a particular problem or situation and to help in interpretation of their data.

The following definitions are provided to familiarize the sampler with the analytical specifications provided in this manual. A proper understanding of these definitions would help minimize the improper use of chemical data.

Range of Application

The range of application described in each method has been chosen to accomodate **the majority of samples in a routine manner**. It is not necessarily a definitive limitation on an analytical technique. Samplers should consult with laboratory staff regarding individual requirements since, in some cases, the specified range may be extended to higher or lower levels.

Detection Criterion, Detection Limit

The criterion and the detection limit are values obtained by statistical manipulation of data collected from each analytical procedure over an extended period of time. These values do not imply any guarantee on the validity or accuracy of any analytical result, but aid the data user in testing the validity of statements which may be made about a given analytical result or environmental situation.

Detection Criterion

The analyst may wish to warn the data user that a given analytical result is so low that there is a reasonable chance that the true value could be zero. The detection criterion is the concentration at or above which the analyst is confident that such a warning is not required.

Detection Limit

If the data user is interested in insuring that the analyst can report positively (i.e. will not report a result lower than the detection criterion) at the environmental concentrations of interest, he makes use of the detection limit. If this value is larger than the anticipated or significant environmental concentration, he may expect to receive a significant number of results from the analyst which do not meet the detection criterion. Thus the risk of concluding that the substance sought is absent when in fact it is present is increased.

The Water Quality Section determines the detection criteria by multiplying the standard deviation of the duplicates in the 0-20% segment of the range by 1.645, and the detection limit by doubling the detection criteria. In doing so, the risk of concluding that a substance is present when it is not or that a substance is absent when it is present, is assumed to be 5%.

The Inorganic Trace Contaminants Section defines the detection limit as being two times the between run standard deviation of samples with low concentrations.

The Pesticides and Organic Trace Contaminant Sections base their detection limits on standard solutions.

Analytical Repeatability and Accuracy

The correctness of a result produced by laboratory staff is limited by many factors, which include the care taken in sampling, the stability of the sample, sample handling and preservation, and analytical variability.

The **repeatability** or precision of the analysis is measured in units of standard deviation, which are determined by analyzing the same sample several times. Each result will be slightly different, but they will all tend to fall within a narrow range of values centered on their average. Under certain circumstances, it can be assumed that the next replicate result will also fall within this range and, in fact, fewer than 5% of the replicates are expected to fall outside of two standard deviations on either side of the average value (fewer than 1% are expected to fall outside of three standard deviations). If only one analysis is performed, there is no way to tell whether it is one of the 5 in 100 results outside of two standard deviations.

Note that repeatability (measured by the standard deviation) is a function of the technique used under routine conditions of operation. If better performance is required by the data user, he should communicate with the appropriate analyst to determine if this is possible within the operational constraints of the laboratory.

Unfortunately, repeatability does not in itself guarantee that the individual (or average) result is "on target". Different analysts tend to introduce their own bias which may change from day to day. Control measures are routinely employed at the various laboratories to ensure data quality, but sampling, sample handling and perishability may all have a significant effect on the accuracy of the results. Both sampler and analyst must work together to ensure that the best quality data is provided for the task at hand. The interpretive value of analytical data is greatly dependent upon precision or repeatability since it is the fundamental criteria for distinguishing between real changes in chemistry and analytical variability.

The precision of a method will vary somewhat over its routine operating range. Therefore, the precision is most correct when reported at a particular concentration level. Some of the precision data listed in the manual will be an average value for an entire range while some will be for a specific concentration level. These data will be coded in the text.

Many of the outlines for pesticides and organic analyses will contain only a general statement of the precision of the test. This was done because these tests may measure several substances and the precision values for these substances may vary widely. For more detailed information, the reader is requested to contact the laboratory personnel in charge of these tests.

Several outlines for trace metals will show precision data, specified at levels outside of the normal operating range. These results will be based on sample dilutions.

The standard deviations quoted for Water Quality Section data are based on within-run duplicates and are broken down into three ranges; less than 20%, 20 to 50%, and 50 to 100% of scale.

Summary

The operating range, detection limit, detection criteria and precision for a particular parameter greatly depend on the type of sample being analyzed, and the methods of sample preparation and analysis used. Open water samples (unpolluted rivers and lakes) can be analyzed to lower detection limits and to better precision than Domestic and Industrial Waste samples. Specifications for all standard operational ranges are presented in the individual outlines.

It is important that the users of the data generated by this laboratory have some understanding of the analytical methods to permit proper interpretation of the results.

NOTE: The methods and statistical data shown in these outlines are for the Toronto Laboratory only. Information on tests done by the regional laboratories is available by contacting them directly.

OUTLINES

ACIDITY

The acidity of a water is a measure of its capacity to neutralize a base or to donate protons. Acidity is defined as the amount of base, expressed as mg/L of calcium carbonate needed to raise the pH of a measured portion of sample to 8.3

The major component of acidity in natural waters is carbonic acid which is formed by the absorption of carbon dioxide. Other lesser components include: portions of other weak acids such as phenolic acid or undissociated tannic acid; hydrolyzable metallic salts, particularly iron and aluminum; and mineral acids which cause an unusually low pH. Acidity is a capacity function and can only be interpreted in terms of specific substances when the chemical composition of the sample is known. Carbonic acid acidity is the predominant form between pH 4.5 and 8.3. Below pH 4.5, mineral acidity predominates.

The acidity of natural waters is increased by industrial activity. Drainage from mine tailings may contain sulphuric acid and hydrolyzable iron due to the aerobic oxidation of pyrite by sulphur bacteria. Oxides of sulphur and nitrogen emitted to the atmosphere from industrial sources contribute to acidic precipitation.

Acid waters inhibit biological activity and have objectionable corrosive properties.

Sampling

Samples should be collected in polystyrene or hard glass containers. Bottles should be filled completely so that no air bubbles remain. Samples should be refrigerated but not frozen and transported to the laboratory as soon as possible. Soil samples should be collected in pomade jars.

Method

Most precipitation, and surface water samples are titrated automatically to a fixed pH endpoint of 8.3 with a standardized sodium hydroxide solution. A few river and industrial waste samples with suspected high buffering capacities are titrated manually. Sediment and soil samples are also titrated manually.

Any substance which can be titrated by a base, is measured by this test.

Time of Analysis

Approximately 60 manual and 30 automatic acidity titrations can be performed per day.

ACIDITY	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	100 mL	50 mL	100 mL	50 mL	50 mL		100 grams					
Preferred Sampling Container	Pyrex or Poly-styrene	Pyrex or Poly-styrene	Pyrex or Poly-styrene	Glass bottle	Glass bottle		Pomade jar					
Preservation Technique	4°C	4°C	4°C	4°C	4°C		4°C					
Operating Range	0.1 to 50	0.1 to 50	0.1 to 20	0.1 to 50	0.1 to 50		*					
Limit Detection Criteria	0.49	0.49	0.26	0.49	0.49							
	0.25	0.25	0.13	0.25	0.25							
Std. Dev. for in run Duplicates	0.15	0.15	0.079	0.15	0.15							
	-	-	0.063	-	-							
	-	-	-	-	-							
Precision												
Units	mg/L as CaCO ₃	mg/L as CaCO ₃	mg/L as CaCO ₃	mg/L as CaCO ₃	mg/L as CaCO ₃		mg/g as CaCO ₃					

* Test available upon request

ALDEHYDES, KETONES

Aldehydes and ketones may impart a disagreeable odour to aqueous solutions or soils. The samples are usually taken from landfill sites and dumping grounds. The substances generally scanned are acetaldehyde, methyl-ethyl ketone and methylisobutyl ketone.

Vapours may irritate the eyes and respiratory systems and may cause delirium. Liquids irritate the skin and eyes. Large doses of acetaldehyde may cause respiratory paralysis. The maximum allowable exposure per 8 hour period is 200 ppm for acetaldehyde and MEK, and 100 for MIBK.

Sampling

Aqueous samples should be collected in 1 liter glass bottles with foil lined caps. Solid samples should be collected in pomade jars. Aqueous and solid samples should be kept cool and transported to the laboratory as quickly as possible.

Method

Aqueous samples are generally analyzed by gas chromatography after direct injection. Solid samples are subjected to head space analysis of the vapours in a sealed bottle or to extraction with an organic solvent prior to gas chromatography.

ALDEHYDES KETONES	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	1000 mL	1000 mL		1000 mL	1000 mL	20 grams	20 grams	20 grams	20 grams			
Preferred Sampling Container	Glass bottle	Glass bottle		Glass bottle	Glass bottle	Glass bottle	Pomade jar	Pomade jar	Pomade jar			
Preservation Technique	4 °C	4 °C		4 °C	4 °C	4 °C	4 °C	4 °C	4 °C			
Operating Range	>1.0	>1.0		>1.0	>1.0	>1.0	>1.0	>1.0	>1.0			
Limit Detection Criteria	1.0	1.0		1.0	1.0	1.0	1.0	1.0	1.0			
Std. Dev. for 20-50% in run 50-100% Duplicates												
Precision	*	*		*	*	*	*	*	*			
Units	mg/L	mg/L		mg/L	mg/L	µg/g	µg/g	µg/g	µg/g			

* = Replicate analyses should be within 10% of the mean

ALIPHATIC ALCOHOLS

Methanol is used as a raw material in the synthesis of formaldehyde and methyl chloride. It is used in solvent dyes, fats, oils and resins. It has been used as a fuel for aircraft, boat and automotive engines.

Ethanol is used in the production of ethyl ether, ethyl esters and chloroform. It is used as an extractant in the manufacture of lacquers, enamels, stains and pharmaceuticals. Most ethanol used for industrial purposes is in the denatured form.

Isopropyl alcohol is an excellent solvent for gums, oils, rosins and some resins. It is used in antifreeze, germicides and liniments and antiseptic solutions. n-Propyl alcohol is used as a solvent for coatings, films, waxes and soaps. It is used as a degreaser for metals and in some medium-duty break fluids.

Amyl alcohols are used as solvents for resins, diluents for hydraulic fluids and as intermediates in organic synthesis. Amyl alcohol vapours may irritate the eyes and respiratory system.

Ethanol is an intoxicant. Butanol vapours irritate the eyes and respiratory system. Liquids, if swallowed, may cause headache, dizziness and narcosis. n-Propanol liquid and vapour irritate the eyes and respiratory system and may be a narcotic at high concentrations. Isopropanol liquid and vapour can cause headaches, dizziness, nausea, narcosis and coma. The fatal dose is about 100 mL of the liquid. Amyl alcohol liquids, if swallowed, may cause headaches, nausea, vomiting and even coma.

The maximum allowable exposures per 8 hours are 400 ppm for isopropanol, 200 ppm for n-propanol, 100 ppm for n-butanol, 1000 ppm for ethanol and 200 ppm for methanol.

Sampling

Aqueous samples should be collected in 1 liter glass bottles with foil lined caps. They should be kept cool and should be transported to the laboratory as quickly as possible. Solid samples should be collected in pomade jars and air samples should be collected in polyester-protected aluminized bags. Aqueous and solid samples should be kept cool and transported to the laboratory as quickly as possible. Bags should be maintained at room temperature.

Method

Samples are generally analyzed by gas chromatography following direct injection.

ALIPHATIC ALCOHOLS	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	1000 mL	1000 mL		1000 mL	1000 mL	20 grams	20 grams	20 grams	20 grams			Half full bag
Preferred Sampling Container	Glass bottle	Glass bottle		Glass bottle	Glass bottle	Glass bottle	Pomade jar	Pomade jar	Pomade jar			Sampling bag
Preservation Technique	4°C	4°C		4°C	4°C	4°C	4°C	4°C	4°C			Keep at room temp.
Operating Range	>1.0	>1.0		>1.0	>1.0	>1.0	>1.0	>1.0	>1.0			>1.0
Limit Detection Criteria	1.0	1.0		1.0	1.0	1.0	1.0	1.0	1.0			1.0 to 10
Std. Dev. for in run Duplicates												
Precision	*	*		*	*	*	*	*	*			*
Units	mg/L	mg/L		mg/L	mg/L	mg/g	mg/g	mg/g	mg/g			mg/m ³

* = Replicate analyses should be within 10% of the mean

ALKALINITY

The alkalinity of a water is a measure of its capacity to neutralize acid. Bicarbonate and carbonate ions are generally the major contributors to the alkalinity of natural water but any ion entering into a neutralisation reaction with strong acid may contribute to the alkalinity as long as the reaction takes place above the pH of the specified endpoint. The hydrolyzable equivalents of silicates, phosphates, arsenates, aluminates and cyanides may all contribute. Hydroxides, which may be present in waters which have been softened by the lime or lime-soda process or have been in contact with cement, may also contribute to the alkalinity.

The alkalinity of a water is generally used to assess the buffering capacity or the water's ability to resist a change in pH. This means that if an acidic waste is discharged into a natural water system, the effect on the water may not necessarily be a decrease in the pH, but may be detected as a drop in the alkalinity.

A fixed endpoint alkalinity (FEA) represents the quantity of acid, required to reduce the pH of a measured portion of sample to pH 4.5. A more precise alkalinity is calculated using the inflection point (IP) of the titration curve.

Waters with an IP alkalinity below 15 mg/L of calcium carbonate are considered to be very poorly buffered.

Sampling

Samples with low alkalinities (below 30 mg/L CaCO_3) should be collected in polystyrene or hard glass containers. Bottles should be filled so that no air bubbles remain. Regular 1 liter glass bottles can be used if the alkalinity is higher than 30 mg/L. Samples should be refrigerated but not frozen. Immediate delivery to the laboratory is essential. Soil samples should be collected in pomade jars.

Method

River and Domestic water samples are titrated automatically to an endpoint of pH 4.5 with a standardized sulphuric acid solution. Precipitation samples are subjected to a Gran analysis of the titration data. Low level river samples are analyzed by a Gran plot technique. Particulates may not be titrated during the reaction time of the titration

Time of Analysis

Approximately 150 surface water, 100 Domestic Water and 30 Precipitation samples may be analyzed per day.

ALKALINITY	Surface Water	Drinking Water	Precip. & low level rivers	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	50 or 100 mL	75 mL	100 mL	75 mL	75 mL		100 grams					
Preferred Sampling Container	Pyrex or poly-styrene	Pyrex or poly-styrene	Pyrex or poly-styrene	Glass bottle	Glass bottle		Pomade jar					
Preservation Technique	4°C	4°C	4°C	4°C	4°C		4°C					
Operating Range	0.7 to 200	1.1 to 400	0.01 to 25.0	1.1 to 400	1.1 to 400		*					
Limit Detection Criteria	1.44	2.23	0.16	2.23	2.23							
	0.72	1.12	0.08	1.12	1.12							
Std. Dev. for in run Duplicates	0.439 0.552 0.586	0.68 0.98 1.70	0.05 - -	0.68 0.98 1.70	0.68 0.98 1.70							
Precision												
Units	mg/L as CaCO ₃	mg/L as CaCO ₃	mg/L as CaCO ₃	mg/L as CaCO ₃	mg/L as CaCO ₃		mg/L as CaCO ₃					

* Test available upon request

ALUMINUM

Aluminum is one of the most abundant elements in nature and is widely distributed in both soluble and insoluble forms. The native metal and its oxide are insoluble, but many salts are highly soluble. Its most important sources include bauxite, cryolite, alum, corundum and kaolin minerals. Aluminosilicate minerals are abundant in igneous and sedimentary rocks and soils. As the minerals weather, aluminum may be left as an insoluble residue such as bauxite. Aluminum compounds occur naturally in water, but may also be derived from industrial wastes or effluents from water treatment plants where alum is commonly used as a precipitating or flocculating agent. Aluminum concentrations are generally less than 1.0 mg/L; however, acid mine drainage may lead to increased levels in certain areas. While aluminum is amphoteric, it is seldom found in the anionic form.

Aluminum is used extensively in domestic utensils, wire, exterior building finishes and in any industrial applications where a strong, light, easily constructed material is needed. Aluminum is also used in dye and paper manufacturing and in the tanning industry. The presence of small amounts of aluminum in water may interfere with certain industrial processes.

Oral toxicity of aluminum and aluminum salts seems to be negligible although ingestion of large amounts seems to alter the body's method of inorganic phosphate elimination. Inhalation of the element or its salts may cause lung problems. Toxic effects on plants have been reported for extreme conditions at both ends of the pH scale, but at a neutral pH, toxicity effects are negligible. Since aluminum has not been shown to be harmful to human health, no drinking water guidelines have been proposed. A limit of 0.100 mg/L has been proposed to protect aquatic biota. The Water Quality Criteria for livestock watering is 5.0 mg/L.

Sampling

Special acid washed plastic containers are required for trace level determinations in domestic water and surface water samples. Glass bottles are required for domestic waste and industrial waste samples. Add 20 drops of HNO_3 per liter. Glass jars are used for soil, sediment and vegetation samples. Vegetation samples should be refrigerated.

Method

Aqueous samples may be preconcentrated before digestion with mineral acids. Solids samples are also digested with mineral acids. Sewage, sludge and Hi-Vol filters are analyzed using ICP atomic emission. All other matrices are analyzed using atomic absorption.

Time of Analysis

The time of analysis depends on the amount of preparation required. Approximately 50 aluminum determinations may be performed per day for aqueous samples.

ALUMINUM	Ground & Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	400 mL	400 mL	25 mL	50 mL	100 mL	10 mL	2 grams	2 grams	50 grams		1 filter strip	
Preferred Sampling Container	Acid-washed plastic bottle	Acid washed plastic bottle	Acid washed plastic bottle	Glass bottle	Glass bottle	Glass bottle	Pomade jar	Plastic bag	Plastic bag		Envelope	
Preservation Technique	HNO ₃ pH <2	HNO ₃ pH <2	HNO ₃ pH <2	HNO ₃ pH <2	HNO ₃ pH <2	None	None	4°C	Freeze		None	
Operating Range	0.002 to 1.0	0.002 to 1.0	0.005 to 0.200	0.08 to 300	0.02 to 10.0	0.40 to 1500	10 to 20,000	20 to 1000	0.25 to 125		0.2004 to 1.5	
Limit Detection Criteria	0.002	0.002	0.005	0.08	0.02	0.40	10	20	0.25		0.2004	
Std. Dev. for 20-50% in run 50-100% Duplicates												
Precision	0.15 at 1.00	0.15 at 1.00	0.005 at 0.139	*	0.15 at 2.00	*	740 at 6,000	6 at 100	-		*	
Units	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	μg/g	μg/g	μg/g		μg/m ³	

* = ± 10% at 10 times the detection limit.

ANIONIC DETERGENTS

(Methylene Blue Active Substances)

Anionic detergents are generally sodium salts of complex organic compounds of the sulphate and sulphonate variety. All are readily soluble in water and ionize to produce the sodium cation and a negatively charged surface active organic ion. They are commonly referred to as "surfactants" because of their ability to reduce the surface tension of water.

Anionic detergents are not found in nature; therefore the presence of these substances in natural waters indicates contamination by domestic or industrial wastes. While generally considered non-toxic to most life forms at low levels, they are objectionable because they cause excessive foaming and inhibit the normal exchange of atmospheric oxygen with water.

The test for anionic detergents is more accurately referred to as "methylene blue active substances" (MBAS) since any material which forms a colored complex with methylene blue will be detected. Anionic surfactants detected include linear alkyl benzene sulphonates (LAS), alkyl sulphates (AS) and alcohol ethoxy sulphates. As the reported result probably represents an overestimate of the anionic detergent concentration, caution should be exercised in interpreting the data. Samples which do not produce any foam when shaken contain less than 0.5 mg/L of MBAS as LAS. Since this is the objective for natural waters, it is not necessary to request a MBAS analysis on samples when a perceptible foam does not appear. The MBAS test does not detect other types of detergents, therefore a special request for an identification should be requested.

Sampling

MBAS are unstable and will be degraded rapidly by bacterial activity. Refrigeration is helpful and immediate delivery to the laboratory is essential. Care must be taken to avoid contamination of the sampling containers by any detergent product used for cleaning.

Method

Samples are mixed with a methylene blue reagent and any substances in the sample which are methylene blue active will be extracted as blue colored salts in chloroform (they are more soluble in chloroform than in water). The chloroform layer is then analyzed colorimetrically using LAS as the calibration standard. Results are reported in mg/L of MBAS as LAS. Reference linear alkyl sulphonate (LAS) solutions with a mean molecular weight of 318 and an activity of 5.55% are prepared by the U.S. Environmental Protection Agency in co-operation with the Soap and Detergent Association.

Time of Analysis

Approximately 36 MBAS analyses may be completed per day.

ANIONIC DETERGENTS (MBAS)	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	100 mL	100 mL		100 mL	100 mL	100 mL						
Preferred Sampling Container	Glass Bottle	Glass Bottle		Glass Bottle	Glass Bottle	Glass Bottle						
Preservation Technique	4 °C	4 °C		4 °C	4 °C	4 °C						
Operating Range	0.1 to 10.0	0.1 to 10.0		0.1 to 10.0	0.1 to 10.0	0.1 to 10.0						
Limit Detection	0.20	0.20		0.20	0.20	0.20						
Criteria	0.10	0.10		0.10	0.10	0.10						
Std. Dev. for in run Duplicates	0.061 0.107 0.151	0.061 0.107 0.151		0.061 0.107 0.151	0.061 0.107 0.151	0.061 0.107 0.151						
Precision												
Units	mg/L LAS	mg/L LAS		mg/L LAS	mg/L LAS	mg/L LAS						

ANTIMONY

Antimony is not generally found in the native state, its major forms in the natural environment are as insoluble sulphides such as the mineral stibnite and oxides such as the minerals cervantite and valentinite.

Antimony is generally found in only trace amounts in natural waters as any dissolved antimony is rapidly adsorbed onto suspended material or precipitated from solution as an oxide. The major sources of antimony in waters are mine wastes, weathering of rock and industrial effluents.

Pure antimony metal is a poor conductor of heat and electricity and is used in the semiconductor industry. The most important use of antimony is as an alloying ingredient for imparting hardness and stiffness to other metals. Batteries, anti-friction alloys, type metal and cable sheathing use a large proportion of the metal produced. Paints, ceramic tile, glass, rubber and pottery are other industries using antimony.

Antimony and several of its compounds are known to be toxic to man. It accumulates in the liver and is detrimental to the heart. Drinking water objectives have not been set. Exposure should be limited to 0.5 mg/m^3 based on a 40 hour work week. The 96 hour LC 50 for fish ranges from 9 to 80 mg/L, depending on the water hardness.

Sampling

Aqueous samples are collected in glass bottles, soil and sediment samples in pomade jars and vegetation in perforated plastic bags. Vegetation samples must be refrigerated.

Method

The sample is digested with an appropriate acid mixture and analyzed by flameless atomic absorption.

Time of Analysis

Approximately 50 antimony analyses can be performed per day.

ANTIMONY	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Vegh	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	50 mL	50 mL					2 grams	0.2 grams				
Preferred Sampling Container	Glass bottle	Glass bottle					Pomade jar	Plastic bag				
Preservation Technique	None	None					None	4°C				
Operating Range	0.001 to 1.0	0.001 to 1.0					*	0.025 to 2.5				
Limits Detection Criteria	0.001	0.001						0.025				
Std. Dev. for 20-50% in run 50-100% Duplicates												
Precision	0.001 at 0.02 mg/L	0.001 at 0.02 mg/L						0.02 at 1.0 µg/g				
Units	mg/L	mg/L					µg/g	µg/g				

* Test available upon request

AROMATIC HYDROCARBONS

Compounds detected in this class include, benzene, toluene, styrene, ethyl benzene, ortho-, meta- and para-xylene, n-propyl benzene, cumene, mesitylene and para-cymene.

Benzene is derived from the coking of coal and from the production of illuminating gas from coal. Benzene is used for blending with gasoline and the manufacture of nylon goods, solvents, varnishes, lacquers, dyes and linoleum. Toxic effects include irritation of mucous membranes, convulsion, bone marrow depression and aplasia.

Toluene is obtained mostly from tar oil. It is used mainly as a solvent. Toluene may be a narcotic at high concentrations and may cause mild macrocytic anemia. It is less toxic than benzene.

Styrene is used in the manufacture of plastics, synthetic rubber, and resins. It may be irritating to the eyes and mucous membrane and may be a narcotic at high levels.

Xylenes are obtained from coal tar and are used mainly as solvents and in the manufacture of dyes and other organics.

The maximum allowable exposures per 8 hours are 200 ppm for toluene, 100 ppm for xylene and styrene, and 1 ppm for benzene. Several of these compounds can be accumulated in fish up to 1000 times that of the ambient concentration within 48 hours. The 96 hour LC 50 levels for fish are 100 mg/L for benzene and styrene and 10 mg/L for toluene and xylene.

Sampling

Aqueous samples should be collected in 1 liter glass bottles with foil lined caps. Solid samples should be collected in pomade jars and air samples should be collected in polyester-protected aluminized bags. Aqueous and solid samples should be kept cool and transported to the laboratory as quickly as possible. Bags should be kept at room temperature and in darkness.

Method

Aqueous samples are generally analyzed by gas chromatography following direct injection. Solid samples are analyzed using head space analysis on the vapours in sealed bottles fitted with "Teflon" septa. Air samples may be analyzed by gas chromatography after direct injection (air bags), thermal desorption (Tenax traps) or by carbon disulphide elution (charcoal traps).

AROMATIC HYDROCARBONS	Surface Water	Drinking Water	Precip. Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	1000 mL	1000 mL	1000 mL	1000 mL	20 grams	20 grams	20 grams	20 grams			Half full bag
Preferred Sampling Container	Glass bottle	Glass bottle	Glass bottle	Glass bottle	Glass bottle	Pomade jar	Pomade jar	Pomade jar			Sampling bag
Preservation Technique	4°C	4°C	4°C	4°C	4°C	4°C	4°C	4°C			Keep in dark place
Operating Range	>1.0	>1.0	>1.0	>1.0	>1.0	>1.0	>1.0	>1.0			>100
Limit Detection Criteria	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0			100
Std. Dev. for in run Duplicates											
Precision	*	*	*	*	*	*	*	*			*
Units	µg/L	µg/L	µg/L	µg/L	µg/g	µg/g	µg/g	µg/g			µg/m ³

* = Replicates should be within 10% of the mean.

ARSENIC

Arsenic is commonly found in nature as the minerals realgar, orpiment, arsenolite and arsenopyrite. The two most common states are As(III) and As(V). Arsenic(V), as arsenate (AsO_4^{3-}) is the stable form in aerobic waters. Arsenite may be present if oxidation to As(V) is incomplete. Arsenic(III) will be the predominant form under anaerobic conditions. In Canada, the dissolved arsenic concentration in river samples collected between 1972 and 1977 ranged from 0.001 to 0.050 mg/L. Lake values varied from less than 0.005 to 0.620 mg/L, Great Lakes from less than 0.0001 to 0.0012 mg/L, sediments from 0.5 to 14.0 mg/kg and fish from 0.03 to 0.12 mg/kg.

Arsenic may be present in natural waters by dissociation of minerals, industrial discharges and pesticide applications. Common industrial applications include the processing of metal ores, glassware, ceramics and tannery products. Manufactured products containing arsenic include wood preservatives, herbicides and insecticides.

As a pollutant in water, arsenic is commonly found as arsenite and arsenate, as the gas arsine, and as organic arsenicals such as methylarsenate, phenylarsenate and trimethyl arsine.

The element and its inorganic salts are highly toxic to humans and are known to exhibit carcinogenic properties. Since arsenic is eliminated from the body very slowly, it tends to accumulate, concentrating in the hair, skin and nails. Arsenic should not be present in domestic waters at concentrations higher than 0.01 mg/L of As. Levels should not exceed 0.1 mg/L to protect aquatic life.

Sampling

Glass bottles are used for water, sewage, industrial waste and sludge samples. Vegetation samples may be collected in perforated plastic bags and refrigerated. Soil and Sediment samples may be collected in pomade jars.

Method

Samples in Toronto are measured by generation of arsine gas and measurement by atomic absorption. Samples in Thunder Bay are measured colorimetrically using silver diethyldithiocarbamate. High concentrations of Se, Sb, Sn, Co, Ni, Cu, Mo and Pt may cause erroneous results in both methods.

Time of Analysis

Approximately 50 arsenic analyses can be performed per day.

ARSENIC	Ground & Surface Water	Drinking Water	Precip. Water	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	50 mL	50 mL		25 mL	25 mL	2 mL	0.2 grams	0.2 grams	50 grams			
Preferred Sampling Container	Glass bottle	Glass bottle		Glass bottle	Glass bottle	Glass bottle	Pomade jar	Plastic bag	Plastic bag			
Preservation Technique	None	None		None	None	None	None	4°C	Freeze			
Operating Range	0.001 to 1.0	0.001 to 1.0		0.001 to 1.0	0.001 to 3.0	0.025 to 0.5	0.025 to 2.5	0.025 to 2.5	0.125 to 125			
Limits Detection Criteria	0.001	0.001		0.001	0.001	0.025	0.025	0.025	0.125			
Std. Dev. for in run Duplicates												
Precision	0.004 at 0.03	0.004 at 0.03		0.004 at 0.03	0.004 at 0.03	0.02 at 0.25	0.2 at 2.4	0.08 at 1.0	-			
Units	mg/L	mg/L		mg/L	mg/L	mg/L	μg/g	μg/g	μg/g			

Std. Dev. for in run Duplicates
 <20%
 20-50%
 50-100%

ASBESTOS

Asbestos is a generic term for several fibrous silicate minerals of the serpentine and amphibole groups. Chrysotile or white asbestos is the sole species classified in the serpentine group and is by far the most abundant kind of asbestos. The amphibole group includes asbestiform types, namely crocidolite, amosite, anthophyllite, tremolite and actinolite.

Asbestos occurs as an environmental water and air pollutant through both natural causes and human operations. Although erosion of rock formations introduces fibres into bodies of water, modern usage of asbestos in over four thousand industrial and domestic applications has provoked concern over the amount introduced into the environment by the mining and milling of asbestos and the manufacture and use of asbestos products.

Growing concern over the long-term health effects of airborne asbestos has led to the belief that asbestos concentrations in the environment should be carefully monitored.

Sampling

A Hi-Vol sampler, equipped with a transducer and continuous air flow recorder, is fitted with a Nuclepore filter of dimensions 20 x 25 cm and 0.5 μ m pore size. Due to the difficulty in handling the Nuclepore filter under field conditions, installation of the filter in a Hi-Vol cassette or filter holder is carried out in a sheltered environment. The cassette may then be attached to and removed from the Hi-Vol at the given field location. After removal of the filter from the cassette, the filter is placed on a separator sheet (supplied with the filter), with the sample side facing up and both folded in half along the 20 cm width. The entire package is then placed in a glassine envelope and delivered to the laboratory together with the air flow data.

For water samples, a polyethylene bottle of 1 liter capacity is rinsed at least three times with the water from which the sample is to be taken and then filled to one inch from the top of the bottle with the water sample. Samples must be shipped to the laboratory without delay and be protected from freezing.

Method

The particulate material from a representative aliquot of the air or water sample is deposited on a 200 mesh electron-microscope grid which is examined at 20000X magnification.

Fibres are classified, counted and sized and the fibre and mass concentrations of chrysotile and amphiboles are calculated and reported.

Time of Analysis

Each sample requires from 3 to 5 hours or more depending on the total particulate loading of the EM grid.

BARIUM

Barium is commonly found in nature as the minerals barite (barium sulphate) and witherite (barium carbonate). It is also found in igneous rock, primarily feldspar, where it is known to replace potassium. Barium ions in solution are rapidly precipitated by sulphate and carbonate. Most other barium salts are soluble. As a result, only trace levels are normally found. Weathering and industrial discharges are the main sources of barium in natural waters. Industrial application of barium includes the manufacture of metal alloys, paints, pigments, paper, soap, rubber, linoleum, flares, cement and various other products.

Insoluble barium salts are non-toxic. Barium sulphate, for example, is used as a contrast material in the radiological diagnosis of ulcers. Soluble salts of barium, however, are readily absorbed through the skin or gastrointestinal tract and exhibit highly toxic effects on the nervous system if present in sufficient quantity. Symptoms include violent muscular spasms, paralysis and cardiac arrest. Trace quantities of soluble barium are known to accumulate in bones, but are not believed to exhibit a cumulative toxic effect like lead or cadmium. The maximum acceptable concentration of barium in a domestic water supply is 1.0 mg/L. There are currently no guidelines for livestock or irrigation waters. Levels of 1.0 mg/L are considered hazardous for marine organisms. The 96 hour LC 50 levels for invertebrates and fish are 10 - 15 mg/L and 50 - 100 mg/L respectively. Barium can be concentrated from water to aquatic organisms by a factor of about 150.

Sampling

Special acid washed plastic containers are required for trace level determinations in domestic water and surface water samples. Glass bottles are required for domestic waste and industrial waste samples. Add 20 drops of HNO_3 per liter. Pomade jars should be used for soil and sediment samples. Vegetation samples should be placed in plastic bags and refrigerated. Fish samples should be placed in plastic bags and frozen.

Method

Samples are digested with the appropriate acid mixture and analysis is accomplished by atomic absorption spectrophotometry using a nitrous oxide-acetylene flame. The analysis is subject to several positive interferences which reduce the experimental accuracy.

Time of Analysis

Approximately 50 barium analyses may be completed per day.

BARIUM	Ground & Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Vegh	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	400 mL	400 mL		50 mL	100 mL	10 mL	2 grams	2 grams	2 grams			
Preferred Sampling Container	Acid washed plastic bottle	Acid washed plastic bottle		Glass bottle	Glass bottle	Glass bottle	Pomade jar	Plastic bag	Plastic bag			
Preservation Technique	HNO ₃ pH <2	HNO ₃ pH <2		HNO ₃ pH <2	HNO ₃ pH <2	None	None	4 °C	Freeze			
Operating Range	0.002 to 0.25	0.002 to 0.25		0.04 to 5.0	0.02 to 2.5	0.20 to 25.0	2.0 to 500	*	*			
Limit Detection Criteria	0.002	0.002		0.04	0.02	0.20	2.0					
Std. Dev. for in run Duplicates												
Precision	0.026 at 0.20	0.026 at 0.20		0.026 at 0.20	0.026 at 0.20	0.026 at 0.20	3.6 at 26					
Units	mg/L	mg/L		mg/L	mg/L	mg/L	µg/g	µg/g	µg/g			

* Test available upon request

BERYLLIUM

Beryllium is a trace element which does not usually occur in natural waters. It is present primarily in particulate form rather than in dissolved form in all except very acidic conditions. Beryllium chloride, nitrate and sulphate are soluble in water but beryllium carbonate and hydroxide are insoluble. Levels seldom exceed 0.001 mg/L.

Weathering of minerals such as beryl accounts for some of the beryllium in natural waters but the majority comes from industrial discharge.

Beryllium metal has a high strength to weight ratio, a high melting point, high modulus of elasticity, high thermal conductivity and is quite resistant to corrosion. Beryllium copper alloys show many of the same properties. As a result, the metal and its alloys have found widespread use in the automotive, aircraft, spacecraft, missile and electrical industries. Beryllium is also used in X-ray lithography for reproduction of micro-miniature integrated circuits.

Beryllium and its salts are toxic and should be handled with the greatest of care. Exposure to beryllium dust should be limited to $2 \mu\text{g}/\text{m}^3$ over a 40 hour work week. The major toxic hazard potential comes from inhaling beryllium containing fumes and dusts. As yet, there are no drinking water guidelines.

At low levels, beryllium is toxic to plants, since it inhibits photosynthesis. Therefore irrigation waters used continuously should not contain concentrations greater than 0.10 mg/L. Levels of 0.011 mg/L may pose a threat to aquatic organisms in waters with a hardness of less than 75 mg/L of CaCO_3 .

Sampling

Special acid washed plastic containers are required for trace level determinations in domestic water and surface water samples. Glass bottles are required for sewage and industrial waste samples. Add 20 drops of HNO_3 per liter. Soil samples should be collected in pomade jars while vegetation samples should be collected in plastic bags and kept refrigerated. Fish samples should be placed in plastic bags and frozen.

Method

The sample is digested with an appropriate acid mixture and analyzed by atomic absorption.

Time of Analysis

Approximately 50 beryllium analyses can be performed per day.

BERYLLIUM	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	400 mL	400 mL		50 mL	100 mL	10 mL	2 grams	2 grams	50 grams			
Preferred Sampling Container	Acid washed plastic bottle	Acid washed plastic bottle		Glass bottle	Glass bottle	Glass bottle	Pomade jar	Plastic bag	Plastic bag			
Preservation Technique	HNO ₃ pH <2	HNO ₃ pH <2		HNO ₃ pH <2	HNO ₃ pH <2	None	4°C	4°C	Freeze			
Operating Range	0.004 to 0.10	0.004 to 0.10		*	*	*	*	*	*			
Limit Detection Criteria	0.004	0.004										
Std. Dev. for 20-50% in run Duplicates												
Precision	0.005 at 0.050	0.005 at 0.050										
Units	mg/L	mg/L		mg/L	mg/L	mg/L	μg/g	μg/g	μg/g			

* Test available upon request

BIOCHEMICAL OXYGEN DEMAND (BOD₅)

The BOD₅ test is a measure of the dissolved oxygen required for the chemical and the aerobic bacterial oxidation of carbonaceous and nitrogenous substances in a five day period at 20°C in the absence of light.

The most frequent damage caused by the discharge of organic wastes to natural waters, is the reduction of dissolved oxygen concentrations to levels which cannot support normal aquatic life. This oxygen depletion arises via the bacterial oxidation of carbon and nitrogen materials and eventually leads to a general deterioration of water quality for all uses. Ideally the BOD₅ should be supplemented by on-site dissolved oxygen measurements in order to assess the effects of stream conditions which offset or accentuate the utilization of dissolved oxygen. The five day period for analysis is arbitrary but has become a standard. Longer periods which include daily oxygen demand measurements are used to measure rates of oxidation and the ultimate demand (k-rates).

The precision of the method is seriously reduced if the sample contains large fragments of suspended solids or an immiscible layer of oil. Representative portions of such samples cannot be properly aliquoted and the results are not generally reproducible within acceptable limits. Biocides such as trace metals seriously interfere with test procedure and samples such as tailing effluents should not be analyzed for BOD.

Sampling

The BOD₅ is a perishable quantity since it is readily influenced by bacterial activity. Immediate delivery to the laboratory is essential. Samples should be collected in 1 L glass bottles and should be kept refrigerated.

Method

Dilutions are prepared on the original sample as necessary, and the dissolved oxygen depletion is measured after a five day incubation period in a sealed bottle at 20°C. Only those dilutions which give a dissolved oxygen depletion between 25 and 75% are accepted for calculating the BOD₅. Dissolved oxygen levels are measured using a membrane electrode which is calibrated via the Winkler Method. Samples with high chlorine residuals and adverse pH levels are chemically adjusted to remove these toxic influences and are then seeded with sewage. Techniques are available for the suppression of the nitrogen-oxygen demand. These can be performed upon special request.

Time of Analysis

By definition, the BOD₅ test will take five days to complete. However, approximately 250 BOD₅ analyses may be set up per day.

BIOLOGICAL OXYGEN DEMAND	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	500 mL	500 mL		500 mL	500 mL							
Preferred Sampling Container	Glass bottle	Glass bottle		Glass bottle	Glass bottle							
Preservation Technique	4°C	4°C		4°C	4°C							
Operating Range	<20	<20		<100	<100							
Limit Detection Criteria	0.34	0.34		2.60	2.60							
	0.17	1.17		1.30	1.30							
Std. Dev. for in run Duplicates	0.102 - -	0.102 - -		0.79 2.03 3.84	0.79 2.03 3.84							
Precision												
Units	mg/L oxygen	mg/L oxygen		mg/L oxygen	mg/L oxygen							

BORON

Boron occurs in nature as the minerals tourmaline, borax, colemanite, boronatocalcite and boracite. These mineral deposits may significantly increase the boron concentration in natural waters. Granites, pegmatites, gabbros and serpentine rocks may also contribute significant amounts of boron by weathering. Boron in water can exist as undissociated boric acid, borate ion and as various polyanionic species. Boron can also enter the waterways through cleaning compounds and industrial waste effluents. Surface water concentrations average about 0.1 mg/L but concentrations may be much higher if groundwater inputs make up a large portion of that water.

Boron is used as a neutron absorber and shielding material in nuclear power plants, in the manufacture of glass, porcelain enamels, detergents, pharmaceuticals, abrasives and dyes. It is also used in fireproofing and waterproofing materials as well as in bactericides, herbicides and fungicides.

Boron is an essential plant nutrient at low levels but high levels may have harmful effects. The maximum permissible concentration of Boron in irrigation waters is 0.75 mg/L. Boron does not appear to accumulate to any extent in the human body. Large doses may cause digestive problems and affect the nervous system. The maximum allowed concentration in irrigation waters is 5.0 mg/L. Levels above 5.0 mg/L may constitute as hazard to some marine organisms.

Sampling

Glass containers should not be used since boron is a major component of glass.

Method

Boron is determined colorimetrically using Azomethine H or quinalizarin for terrestrial and vegetation samples. A preliminary cleanup, ashing or fusion is performed depending on the sample matrix. Hot water extraction is commonly used for soils and sediments. High concentrations of humates, iron, aluminum, zinc and copper may interfere with the Azomethine H method.

Time of Analysis

Approximately 20 boron determinations may be performed per day.

BORON	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	50 mL	50 mL		50 mL	50 mL	50 mL	2 grams	20 grams				
Preferred Sampling Container	Plastic bottle	Plastic bottle		Plastic bottle	Plastic bottle	Plastic bottle	Plastic bag	Plastic bag				
Preservation Technique	None	None		None	None	None	None	4°C				
Operating Range	0.02 to 3.0	0.02 to 3.0		0.02 to 3.0	0.02 to 3.0	0.04 to 6.0	*	0.10 to 30				
Limit Detection Criteria	0.02	0.02		0.02	0.02	0.04		0.10				
Std. Dev. for in run Duplicates	0.02 at 0.2	0.02 at 0.2		0.02 at 0.2	0.02 at 0.2	0.04 at 0.4		1 at 10				
Precision	0.02 at 0.2	0.02 at 0.2		0.02 at 0.2	0.02 at 0.2	0.04 at 0.4		1 at 10				
Units	mg/L	mg/L		mg/L	mg/L	mg/L	μg/g	μg/g				

* Test available upon request

CADMIUM

Cadmium levels in natural waters are generally below 0.01 mg/L unless there is an industrial source nearby. While weathering of rocks and minerals such as greenockite (cadmium sulphide) adds some cadmium to waters the principal sources are industrial processes. Since greenockite is often found with zinc sulphide ore, the mining and smelting of zinc, lead and copper release large amounts of cadmium to the air and water. It may be discharged from the manufacturing of nickel cadmium batteries, various alloys, solders, ceramics, pigments and printing inks. It is also used in some pesticides and fertilizers. The burning of fossil fuels accounts for a large part of the cadmium emissions.

All forms of cadmium are highly toxic, and once ingested are likely to remain in the body for a long time, becoming concentrated in the liver, kidneys and other organs. Cases have been reported where toxic effects did not become apparent until many years after the last exposure. The water quality criteria for cadmium in domestic water supplies is 0.01 mg/L. The presence of cadmium reduces plant growth. Since it can be accumulated within plants which may be used as a food source, irrigation waters should not contain more than 0.050 mg/L when used on neutral or alkaline soils and 0.010 mg/L when used continuously on all soils. Levels above 0.0002 mg/l may be harmful to some aquatic organisms.

Sampling

When levels below the normal range of application are required, a notation must be included on the sample submission form so that the sample can be preconcentrated prior to analysis. One full 32 oz bottle is required exclusively for the preconcentration process.

Special acid washed plastic containers are required for trace level determinations in domestic water and surface water samples. Glass containers are required for domestic waste and industrial waste samples. Add 20 drops of HNO_3 per liter. Use pomade jars for soil and sediment samples. Vegetation samples should be collected in plastic bags and refrigerated. Fish samples should be placed in plastic bags and frozen.

Method

Samples are digested using the appropriate acid mixture and then analyzed. Sewage, sludge and Hi-Vol samples are analyzed by ICP atomic emission. All other matrices are analyzed by atomic absorption. Polarographic procedures may be used for ultra trace concentrations in some matrices.

Time of Analysis

Approximately 50 analyses may be performed per day.

CADMIUM	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	400 mL	400 mL	25 mL	50 mL	100 mL	10 mL	2 grams	2 grams	50 grams	200 mL	1 filter strip	
Preferred Sampling Container	Acid washed plastic bottle	Acid washed plastic bottle	Acid washed plastic bottle	Glass bottle	Glass bottle	Glass bottle	Pomade jar	Plastic bag	Plastic bag	Sampling jar	Envelope	
Preservation Technique	HNO ₃ pH <2	HNO ₃ pH <2	HNO ₃ pH <2	HNO ₃ pH <2	HNO ₃ pH <2	None	None	4°C	Freeze	None	None	
Operating Range	0.0002 to 0.01	0.0002 to 0.01	0.0001 to 0.03	0.03 to 300	0.02 to 10.0	0.30 to 300	0.3 to 250	0.2 to 100	0.025 to 1.25	0.03 to 3.0	0.0002 to 0.35	
Limit Detection Criteria	0.0002	0.0002	0.0001	0.03	0.02	0.30	0.30	0.20	0.025	0.03	0.0002	
Std. Dev. for in run Duplicates												
Precision	0.015 at 0.20	0.015 at 0.20	0.00004 at 0.00057	*	0.02 at 0.40	*	0.15 at 1.5	0.6 at 10	-	-	*	
Units	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	μg/g	μg/g	μg/g	μg/m ² 30 days	μg/m ³	

* = ± 10% at 10X Detection Limit

CALCIUM

Calcium ranks fifth in the order of natural elemental abundance and is commonly found in deposits of limestone, dolomite, gypsum and gypsiferous shale. It is generally present in all natural waters at levels which depend on the amount of contact with specific geological formations. Typically, levels in waters are less than 15 mg/L but waters in contact with carbonate rocks may contain up to 100 mg/L. For this reason, surface waters generally tend to have lower calcium levels than ground waters. The calcium content may vary from zero to several hundred milligrams per liter depending on the source and treatment of the water.

Calcium enters the waterway through leaching of soils, weathering of rocks, effluents of breweries and glue factories, and from road salt.

Calcium is an essential element for all life forms. It enhances biological productivity in plants and promotes proper bone and teeth formation in animals. Aqueous calcium compounds are generally non-toxic and represent no known health hazard.

While low concentrations of calcium assist in preventing the corrosion of metal pipes by forming a protective layer of calcium carbonate, high concentrations cause excessive scale which lead to restricted flow and pressure build-ups. Calcium is the major contributor to water "hardness".

A limit of 200 mg/L has been proposed for drinking water with an objective of 75 mg/L.

Sampling

Samples should be collected in glass or polystyrene bottles. Non-routine industrial wastes, or any other samples where high levels of heavy metals are suspected, should be collected in plastic containers and preserved with 20 drops of nitric acid per liter. Sludges need no preservation. Soils should be collected in pomade jars. Vegetation samples should be collected in perforated plastic bags and kept refrigerated.

Method

Precipitation and river samples are analyzed by atomic absorption. Domestic water samples are titrated with EDTA to the murexide endpoint. Industrial waste, soil and dustfall samples are analyzed by atomic absorption. Sewage and sludge are analyzed by ICP atomic emission. Most other matrices are analyzed by atomic absorption. For calcium analyses in vegetation by X-ray fluorescence, see Matrix Elements in Vegetation.

Time of Analysis

Approximately 70 domestic water calcium analyses can be performed per day with the titrimetric method and 200 surface water analyses can be performed per day using atomic absorption. Approximately 50 samples per day can be performed for the other matrices.

CALCIUM	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Vegh	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	100 mL	100 mL	100 mL	50 mL	100 mL	10 mL	2 grams	2 grams		200 mL		
Preferred Sampling Container	Glass or poly-styrene	Glass or poly-styrene	Poly-styrene	Glass bottle	Glass bottle	Glass bottle	Pomade jar	Plastic bag		Sampling jar		
Preservation Technique	None	None	4°C	HNO ₃ pH <2	HNO ₃ pH <2	None	None	4°C		None		
Operating Range	0.2 to 20.0	1.3 to 120	0.06 to 2.00	0.1 to 300	0.1 to 300	5.0 to 300	10 to 2500	10 to 1000		0.13 to 2.6		
Limit Detection	0.50	2.63	0.11	0.05	0.1	5.0	10	10		0.13		
Criteria	0.25	1.32	0.056									
Std. Dev. for in run Duplicates	0.151 0.199 0.178	0.80 0.80 0.80	0.034 0.017 0.030									
Precision				*	0.02 at 0.15	*	60 at 1000	5 at 100				
Units	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	μg/g	μg/g		μg/m ² /30 days		

* = ± 10% at 10X the Detection Limit

CARBAMATE INSECTICIDES AND HERBICIDES

This group includes (N-phenyl) herbicides such as sutan, CIPC, ICP, Captan, diallate, barban, benomyl and eptam and (N-methyl) insecticides such as carbofurn, propoxure, aminocarb and bux. Carbamate herbicides are frequently used as pre-emergent treatment for grass seedlings and weed control in vegetable crops. Levels for diallate and barban in raw water and livestock waters must not exceed 100 $\mu\text{g/L}$ and are rated as being slight and high ecological hazards respectively.

Carbamate insecticides behave similarly to organo-phosphorus insecticides, in that they depress cholinesterase activity and affect nerve transmission. Carbofuran levels should not exceed 100 $\mu\text{g/L}$ in raw or livestock water supplies. It has been rated as a moderate ecological hazard. Benomyl is very toxic to fish and invertebrates. The 48 hour LC 50 to fish is less than 10 mg/L. Carbamate insecticides and herbicides should not be found in livestock watering waters at levels exceeding 0.1 mg/L.

Sampling

Samples should be collected in solvent rinsed, brown glass bottles with foil lined caps. Bottles should be filled to the mark, refrigerated and transported to the laboratory as quickly as possible.

Method

Samples undergo solvent extraction, clean-up with Florisil and analysis by HPLC.

Time of Analysis-

Approximately 6 analyses can be performed per day.

CARBAMATES INSECTICIDES AND HERBICIDES	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	800 mL	800 mL			800 mL							
Preferred Sampling Container	Special bottle	Special bottle			Special bottle							
Preservation Technique	4° C	4° C			4° C							
Operating ** Range	>0.10	>0.10			>0.10							
Limit Detection Criteria	0.10 to 0.40	0.10 to 0.40			0.10 to 0.40							
Std. Dev. for in run Duplicates												
Precision	*	*			*							
Units	µ g/L	µ g/L			µ g/L							

* = Replicate analyses should be within 10% of the mean.

** = Actual operating range may vary from the detection limits shown to percent levels.

CARBON DIOXIDE

Dissolved carbon dioxide (carbonic acid) is generally found in all water samples with a pH between 4.5 and 8.3. Surface water contains dissolved CO₂ as a result of weathering, biological activity and atmospheric absorption. Levels seldom exceed 10 mg/L of CO₂. Ground waters and waters from the hypolimnion of stratified lakes and reservoirs which contain dissolved CO₂ as a result of bacterial activity may have concentrations in excess of this level. The carbon dioxide content of a water contributes greatly to its corrosive properties.

Since the dissolved carbon dioxide determination has many sources of error, caution must be exercised in its interpretation. It is an acidity measurement and will include all forms of acidity, not only CO₂. Samples with a pH close to 4.5 may produce CO₂ results which are really the sum of the carbonic acid acidity and a portion of the mineral acidity. Any changes in temperature or barometric pressure are likely to change the CO₂ content of the sample. No water quality standards exist for dissolved carbon dioxide.

Sampling

Special precautions are required to minimize agitation of the sample otherwise CO₂ will be lost. Samples are to be carefully transferred from the sampling device into the bottom of a special leak proof glass stoppered container via a syphon. Allow plenty of sample to overflow before the container is finally capped. If any air bubbles remain in the container, analysis cannot be performed. Samples should then be refrigerated and brought to the laboratory within 24 hours. Samplers generally deliver CO₂ samples personally as they are unsafe to ship commercially, and agitation should be minimized.

Consultation with laboratory staff is required prior to sampling as special preparations are necessary before the samples arrive. Field determinations are recommended when possible.

Method

Samples are transferred by syphon to Nessler tubes where they are titrated potentiometrically with carbonate free sodium hydroxide to a pH endpoint of 8.3. Special precautions are practised to limit any errors due to atmospheric exchange of CO₂. Results are reported in mg/L of CO₂. Mineral acids and salts of strong acids and bases will interfere. High levels of aluminum, chromium, copper, iron, amines, ammonia, borate, phosphate silicate and sulphide may cause high results. This method is not applicable to acid mine wastes.

Time of Analysis

Approximately 40 CO₂ analyses may be completed by titration per day.

CARBON DIOXIDE

Minimum Sample Size Full special bottle

Preferred Sampling Container See text

Preservation Technique None

Operating Range 1.0 to 88

Limit Detection Criteria 1.64 0.82

Std. Dev. for in run Dup. <20% 20-50% 50-100%

Precision

Units mg/L mg/L

Surface Water

Drinking Water

Precip.

Sewage

Ind. Wastes

Sludge

Soil & Sediment

Veg'n

Bio Material

Dust Fall

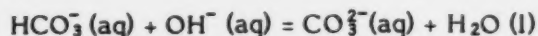
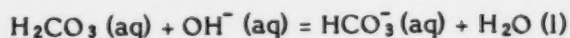
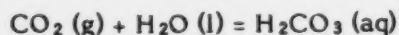
Hi-Vol

Ambient Air

CARBON - DISSOLVED INORGANIC

The dissolved inorganic carbon test measures dissolved carbon dioxide, bicarbonate and carbonate. Inorganic carbon enters the water from the atmosphere and by weathering of carbonaceous rocks such as limestones and dolomites. Bicarbonate salts may also be added by industrial effluent.

Carbonate equilibria in natural water systems are governed by the equations below, where pH, the partial pressure of CO₂ and sample alkalinity are the controlling factors.



Generally, below pH 4.5, H₂CO₃ (aq) is the predominant species, between pH 4.5 and 8.3, bicarbonate is the major species and above pH 8.3, carbonate is the major ion present.

Dissolved inorganic carbon is another measure of the buffering capacity or alkalinity of a natural water and there is a relationship between DIC and alkalinity.

The dissolved inorganic carbon test can be used in estimating primary productivity levels by determining how much dissolved inorganic carbon is available for uptake by algae for photosynthesis.

At present there are no established limits for carbonate or bicarbonate in waters, however high levels may be objectionable due to precipitation with calcium and magnesium to form a scale on metal surface.

Sampling

Typical river and drinking water samples should be collected in glass bottles. Samples for the low dissolved inorganic carbon test should be collected in hard glass (pyrex) tubes with poly seal cone caps and filled to capacity with no air bubbles trapped. Samples should be transferred to the laboratory as quickly as possible. For high levels there is a possibility of loss of CO₂ and deposition of carbonate on the walls of the container and therefore containers should be filled to capacity. Bottles containing sludges should be filled only half full.

Method

Dissolved inorganic carbon is measured colorimetrically by acidifying the sample and allowing the CO₂ produced to pass through a gas permeable membrane into a weakly buffered, alkaline, phenolphthalein solution. The decrease in absorbance of this colored solution is then measured. Chlorine, hydrogen sulphide and humates interfere.

Time of Analysis

Approximately 50 dissolved inorganic carbon analyses can be performed per day.

CARBON DISSOLVED INORGANIC	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	CARBON LOW LEVEL DISSOLVED INORGANIC	Surface Water
Minimum Sample Size	50 mL	50 mL	mL	50 mL	50 mL	50 mL	Full pyrex tube	
Preferred Sampling Container	Glass bottle	Glass bottle		Glass bottle	Glass bottle	Glass bottle	Pyrex tube with polyseal cap	
Preservation Technique	4°C	4°C		4°C	4°C	4°C	4°C	see text
Operating Range	0.3 to 40.0	0.3 to 40.0		0.3 to 40.0	0.3 to 40.0	0.3 to 40.0	0.05 to 2.00	
Limit Detection Criteria	0.54	0.54		0.54	0.54	0.54	0.086	
Std. Dev. for in run Duplicates	0.165 0.213 0.310	0.165 0.213 0.310		0.165 0.213 0.310	0.165 0.213 0.310	0.165 0.213 0.310	0.026 0.061 -	
Precision								
Units	mg/L carbon	mg/L carbon		mg/L carbon	mg/L carbon	mg/L carbon	mg/L carbon	

CARBON - DISSOLVED ORGANIC

Carbon is the basic building block of all life. Inorganic and organic forms of carbon which enter the water from the atmosphere, surrounding lands and basins are interconverted during the natural life cycle.

The organic content of lakes and rivers depends primarily on the products of plants and animals which those water bodies support. Most of the organic carbon in water is composed of humic substances and partly degraded plant and animal materials, some of which is resistant to microbial degradation. Runoff from agricultural land and industrial discharge from industries such as pulp and paper will add organic carbon to the water. The amount of organic carbon produced by photosynthetic processes will vary from season to season. The degradation of large amounts of organic matter causes depletion of the dissolved oxygen concentration and hence, organic carbon is also measured on sewage and industrial waste samples.

In natural waters, the organic carbon content will usually be less than 30 mg/L. This test may also measure some particulate carbon even though precautions are taken to minimize this.

Sampling

Samples should be collected in glass bottles. Samples should be transported to the laboratory as quickly as possible. Sludge samples should be filled only half way.

Method

The dissolved organic fraction is measured after it has been stripped of inorganic carbon and the sample has been oxidized by a U.V. digester in an acid-persulphate medium. The dissolved organic carbon is then measured colorimetrically by allowing the CO₂ produced to pass through a gas permeable membrane into a weakly buffered, alkaline phenolphthalein solution. The decrease in absorbance of this colored solution is then measured.

Time of Analysis

Approximately 50 dissolved organic carbon analyses can be performed per day.

CARBON DISSOLVED ORGANIC	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	50 mL	50 mL	mL	50 mL	50 mL	50 mL	grams	grams	grams	mL		
Preferred Sampling Container	Glass bottle	Glass bottle		Glass bottle	Glass bottle	Glass bottle						
Preservation Technique	4°C	4°C		4°C	4°C	4°C						
Operating Range	0.2 to 20.0	0.2 to 20.0		0.2 to 20.0	0.2 to 20.0	0.2 to 20.0						
Limit Detection	0.49	0.49		0.49	0.49	0.49						
Criteria	0.24	0.24		0.24	0.24	0.24						
Std. Dev. for in run Duplicates	0.148 0.100 0.123	0.148 0.100 0.123		0.148 0.100 0.123	0.148 0.100 0.123	0.148 0.100 0.123						
Precision												
Units	mg/L carbon	mg/L carbon		mg/L carbon	mg/L carbon	mg/L carbon						

CHEMICAL OXYGEN DEMAND (COD)

The chemical oxygen demand is a measure of the oxygen required to oxidize susceptible organic and inorganic compounds under vigorous chemical oxidizing conditions. This test is especially useful on industrial wastes where the BOD₅ test is not applicable. Toxic materials in industrial wastes tend to inhibit bacterial activity producing a BOD₅ which generally underestimates the true organic composition of the sample. Most organic compounds are oxidized by the COD method. Benzene, toluene, pyridine, and similar aromatic ring compounds are incompletely recovered. The COD may be used to estimate the effect of a particular waste on a receiving water or may be used to estimate the efficiency of a waste treatment process.

COD results do not necessarily relate directly to the BOD₅ value or to the oxygen consumption in the receiving water, but they do represent the ultimate carbonaceous oxygen demand that may eventually be exerted. If a number of comparative analyses show that a stable ratio exists between the COD and BOD₅ for a particular waste, then the COD results may be used to predict the approximate BOD₅ values.

Sampling

The COD is a perishable quantity since it may be influenced by bacterial activity. Immediate delivery to the laboratory is essential. Aqueous samples should be collected in 1 L glass bottles and refrigerated. Soil samples should be collected in pomade jars.

Method

A water sample is refluxed with a known amount of potassium dichromate and sulphuric acid for two hours. Mercuric sulphate is added to prevent chloride interference, and silver sulphate is added to catalyze the oxidation of straight chain hydrocarbons. The oxygen utilized by the sample is determined by titration with ferrous ammonium sulphate and results are reported in mg/L of oxygen.

Time of Analysis

Approximately 30 COD analyses may be completed per day.

CHEMICAL OXYGEN DEMAND	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'h	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	25 mL	25 mL		25 mL	25 mL	25 mL	2 grams					
Preferred Sampling Container	Glass bottle	Glass bottle		Glass bottle	Glass bottle	Glass bottle	Pomade jar					
Preservation Technique	4°C	4°C		4°C	4°C	4°C	None					
Operating Range	3 to 1000	6 to 1000		6 to 1000	6 to 1000	*	*					
Limit Detection	5.42	11.0		11.0	1.0							
Criteria	2.71	5.51		5.51	5.51							
Std. Dev. for in run Duplicates	1.65 - -	3.35 5.32 7.30		3.35 5.32 7.30	3.35 5.32 7.30							
Precision												
Units	mg/L Oxygen	mg/L Oxygen		mg/L Oxygen	mg/L Oxygen	mg/L Oxygen	mg/L Oxygen					

* = Test available upon request.

CHLORIDE

Chloride, in the form of the Cl ion, is a major anion in domestic wastes and in many natural water supplies. Urban runoff often contains high concentrations of chloride in winter due to the application of road salt.

Chloride poses no direct health hazard, but the water quality objective for domestic water is 250 mg/L of Cl to prevent a salty taste. This salty taste is variable and depends on the composition of the water. If chloride is present as sodium chloride, a detectable taste may be present at 250 mg/L, but if it is present as calcium or magnesium chloride there may be no noticeable taste at 1000 mg/L. A high chloride content may harm metallic pipes and structures as well as agricultural plants.

The colorimetric and titrimetric methods of chloride analysis actually measure chloride, bromide, iodide and cyanide. Therefore, caution must be exercised in interpreting the results. Generally, in natural waters, the concentrations of bromide, iodide and cyanide are not large enough to cause interference. In regions where watercourses are influenced by ocean tide-waters, or industrial discharge, these ions may provide appreciable interference.

Sampling

Water, sewage, industrial waste and sludge samples should be collected in glass bottles. Precipitation samples should be collected in polystyrene bottles, soil samples in pomade jars and vegetation samples in plastic bags.

Method

Domestic water samples are analyzed by titrating a measured volume of sample with a standardized silver nitrate solution. River lab samples, soils and vegetation are analyzed colorimetrically using mercuric thiocyanate and ferric ammonium sulphate. Bromide, iodide and cyanide interfere with the colorimetric test. Precipitation samples are analyzed by ion chromatography. Dustfall and Hi-Vol samples are measured by ion specific electrode. Vegetation samples may also be analyzed by X-ray fluorescence, but it is elemental chlorine, not chloride, which is reported. (See matrix elements in vegetation).

Time of Analysis

Approximately 120 chloride analyses may be performed per day titrimetrically, 200 colorimetrically and 30 by ion chromatography.

CHLORIDE	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Vegh	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	50 mL	50 mL	50 mL	50 mL	50 mL	50 mL	20 grams	20 grams		100 mL	2 filter strips	
Preferred Sampling Container	Glass bottle	Glass bottle	Polystyrene bottle	Glass bottle	Glass bottle	Glass bottle	Pomade jar	Plastic bag		Sampling jar	Envelope	
Preservation Technique	None	4°C	None	None	None	None	None	4°C		None	None	
Operating Range	0.1 to 10	0.5 to 400	0.03 to 1.5	0.5 to 400	0.5 to 400	0.5 to 100	125 to 25,000	25 to 5,000		0.005 to 3.00	0.3 to 30	
Limit Detection	0.168	1.02	0.056	1.02	1.02	0.5	125	25		0.005	0.3	
Detection Criteria	0.084	0.51	0.028	0.51	0.51							
Std. Dev. for in run Duplicates	<20% 0.051 0.056 0.065	0.31 0.25 0.52	0.017 0.017 -	0.31 0.25 0.52	0.31 0.25 0.52							
Precision						1 at 20	1000 at 20,000	100 at 2,000		-	1.36 at 4.2	
Units	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	μg/g	μg/g		μg/m ² / 30 days	μg/m ³	

CHLORINE - RESIDUAL

Chlorination of water supplies and polluted waters is performed to destroy or deactivate disease producing organisms. The chlorine may also react with ammonia, iron, manganese, sulphide and some organic substances resulting in improved water quality. Chlorination may produce adverse effects by intensifying the taste and odour characteristics of phenols and other organic compounds present in water.

Elemental chlorine is a gas which is highly soluble in water and reacts readily with many inorganic substances and all plant and animal tissues. Liquified chlorine gas assumes three principal forms of free chlorine; hypochlorous acid, hypochlorite ion and aqueous molecular chlorine. Free chlorine reacts readily with ammonia and certain nitrogenous compounds to form combined available chlorines such as mono- and di-chloramine. The presence and concentration of these combined forms depends on pH, temperature and the initial chlorine to nitrogen ratio. While treated waste waters may contain both free and combined forms, the free chlorine fraction is seldom found because it is generally added in a smaller amount than the demand of that water. Total residual chlorine (TRC) is the sum of the free available chlorine and the combined available chlorine.

In many water treatment plants, sodium hypochlorite is being used instead of chlorine gas because it is safer. Chlorine gas can be toxic, cause severe lung irritation and damage, irritate the eyes and can cause conjunctivitis. TRC levels of 0.014 to 0.029 mg/L can be toxic to caged rainbow trout. The Water Quality Objective states that levels should not exceed 0.002 mg/L in surface water because of toxicity to fish.

Sampling

Samples should be taken in glass bottles and filled to the top so that no air bubbles remain and kept refrigerated. Agitation or exposure to sunlight will accelerate the reduction of chlorine. Samples should be analyzed as soon as possible after collection and if possible, samples should be analyzed in the field.

Method

Samples are analyzed by an amperometric titration. TRC, free available chlorine, monochloramine and dichloramine may all be measured by varying the amount, order and type of reagent.

Sewage and drinking water samples may also be analyzed for TRC using the Palin DPD comparator method.

Time of Analysis

Approximately 20 titrations can be performed per day.

CHLORINE RESIDUAL

Minimum Sample Size

Preferred Sampling Container

Preservation Technique

Operating Range

Limit Detection Criteria

Std. Dev. for in run Duplicates

Precision

Units

Surface Water

1000 mL

Glass bottle

See text

$\mu\text{g/L}$ as Chlorine

Drinking Water

1000 mL

Glass bottle

See text

$\mu\text{g/L}$ as Chlorine

Precip.

mL

Sewage

1000 mL

Glass bottle

See text

<4.0

mg/L as Chlorine

Ind. Wastes

Sludge

Soil & Sediment

Veg'n

Bio Material

Dust Fall

Hi-Vol

Ambient Air

CHLOROPHENOL INSECTICIDES

Chlorophenols exhibit germicidal and insecticidal properties and are used as flea repellents, fungicides and wood preservatives. In general, their effectiveness increases with the amount of chlorine substitution. Chlorophenols enter the environment through agricultural runoff and pulp and paper wastes.

At present this test measures 2,4,6 trichlorophenol, 2,4,5 trichlorophenol, 2,3,4 trichlorophenol and penta chlorophenol.

Pentachlorophenol is used primarily as a wood and starch preservative but can be used also as an insecticide for termites, a pre-harvest defoliant and as a general herbicide. The trichlorophenols are often found as impurities in the penta-chlorophenol. The 2,4,5 compound is used as a fungicide and a bactericide. The 2,4,6 compound is used as a fungicide, a bactericide and a preservative.

Toxicity varies from compound to compound. Ingestion of PCP can cause decreased respiration, blood pressure, weakness, convulsions and death. It can also cause lung, liver and kidney damage.

Chlorophenols are extremely toxic to fish with a 96 hour LC₅₀ for fish of less than 1 mg/L. Chlorophenols may also cause taste and odour problems in drinking waters.

At present there are no specified tolerance limits for chlorophenols.

Sampling

Samples should be collected in solvent rinsed, brown glass bottles. Bottles should be filled to the mark, preserved with 5 mL of concentrated phosphoric acid, refrigerated and transported to the laboratory as quickly as possible.

Method

Samples undergo solvent extraction, evaporation, derivatisation, clean-up with florisil and analysis by electron capture gas chromatography.

CHLOROPHENOL INSECTICIDES	Surface Water	Drinking Water	Precip. Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n Material	Bio Material	Dust Fall	Hi-Vol Air	Ambient Air
Minimum Sample Size	800 mL	800 mL		800 mL		10 grams	10 grams	10 grams			
Preferred Sampling Container	Special bottle	Special bottle		Special bottle		Pomade jar see text	Pomade jar see text	Pomade jar see text			
Preservation Technique	5 mL H ₃ PO ₄ 4°C	5 mL H ₃ PO ₄ 4°C		5 mL H ₃ PO ₄ 4°C		4°C	4°C	4°C			
Operating ** Range	>0.05	>0.05		>0.05		0.05	0.05	0.05			
Limit Detection Criteria	0.05	0.05		0.05		-	-	-			
Std. Dev. for 20-50% 50-100% Duplicates											
Precision	*	*		*							
Units	µg/L	µg/L		µg/L		µg/g	µg/g	µg/g			

* = Replicate analyses should be within 10% of the mean.

** = Actual operating range may vary from the detection limits shown to percent levels.

CHLOROPHYLL

Chlorophyll is the natural pigment component of all green plants. It is actively involved in photosynthesis. The quantity of chlorophyll is therefore a good indication of how much plant material is present in a water sample. Chlorophyll data provides a way to estimate the biomass of standing algae crops which can then be used to assess the effectiveness of nutrient removal programs as well as the general trophic status of lakes and rivers.

Chlorophyll results are generally reported as "chlorophyll a", "chlorophyll b", and "acidified chlorophyll a". Generally, the "acidified chlorophyll a" is considered most useful because it represents the pigment contribution of living cells. "Chlorophyll a" represents the pigment contribution of both living and dead cells (degraded chlorophyll pigments in dead cells are called pheophytins). Chlorophyll may constitute up to 1 - 2% of the dry weight. "Chlorophyll b" is often very small relative to "chlorophyll a", therefore its interpretive value is limited. As the analytical variability becomes very large for high concentrations of chlorophyll, caution should be exercised in interpreting results where the "chlorophyll a" exceeds 20 µg/L.

Sampling

Add 5 drops of a 10% $MgCO_3$ suspension to about 1 liter of sample. This prevents the conversion of chlorophyll to pheopigments at low pH levels. Filter a measured aliquot through a 1.2 micron Sartorius CN membrane filter. Place the filter in a labelled petrie dish being sure to include the exact volume of the filtered aliquot on the label. Wrap the petrie dishes in aluminum foil and ship to the Laboratory for analyses.

If filtration in the field is not possible, efforts must be made to retard photosynthetic activity. Samples should be taken in glass bottles, refrigerated and kept in the dark. Immediate delivery to the Laboratory is essential.

Method

Chlorophyll is extracted from the cells and analysis is accomplished spectrophotometrically. The final results are mathematically determined by considering absorbance readings at four different wavelengths using the SCOR-UNESCO equations. Any substance which absorbs light at these wavelengths will be detected as an interference.

Time of Analysis

Approximately 120 samples can be performed over a two day period.

	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
--	---------------	----------------	---------	--------	-------------	--------	-----------------	-------	--------------	-----------	--------	-------------

1000 mL

**Glass
bottle
or field
filtration**

See
Text

20

<20%
20-50%
50-100%

Precision

 $\mu\text{g/L}$

CHROMIUM

Chromium appears in nature primarily as the mineral chromite or chrome iron ore. Chromium in rocks and soils is generally present as insoluble chromic oxide. During weathering, chromium behaves like iron and is retained in sands and clays. Thus little of the chromium actually goes into solution. Natural waters generally contain less than 0.001 mg/L. Chromium(III) and chromium(VI) are the two forms most common in waters. The hexavalent form is the most common since above pH 5, the trivalent ion precipitates out or gets oxidized to the hexavalent form.

Chromium can enter the waterways as a result of weathering or from industrial discharges from cement, ferrochromium, chromium steel and metal plating industries. The burning of fossil fuels also contributes a large amount of chromium.

Chromium is present in small quantities in all soils and plants, but there is no evidence to indicate that it is required by plants for growth since chromic and chromate ions interfere with the uptake of essential elements. Soils with a high chromium content are generally infertile. Hexavalent chromium is the most toxic inorganic form of the element and has been demonstrated to exhibit carcinogenic properties. The maximum permissible concentration in domestic water supplies has been established at 0.05 mg/L of hexavalent chromium. Concentrations of chromium in unfiltered waters should not exceed 0.1 mg/L to protect aquatic life. The maximum allowable concentration of dusts and mists in air, measured as CrO_3 is 0.1 mg/m³ per 8 hour exposure.

Sampling

Fill the sampling bottle completely and deliver it to the laboratory as quickly as possible. Chromate ion is readily adsorbed on container surfaces and delays in shipment longer than 2 to 3 days may cause erroneous results.

Special acid washed plastic containers are required for trace level determinations in domestic water and surface water samples. Glass containers are required for domestic waste and industrial waste samples. Add 20 drops of HNO_3 per liter for total chromium analyses. Pomade jars are used for soil and sediment samples. Vegetation samples should be collected in plastic bags and refrigerated.

Method

Hexavalent chromium samples are analyzed colorimetrically with diphenylcarbazide with no sample pretreatment. High levels of chloride, vanadium, iron and mercury may interfere.

Total chromium samples are digested with the appropriate acid solution and analyzed by atomic absorption or ICP atomic emission (sewage, sludge and Hi-Vol samples).

Time of Analysis

Approximately 50 atomic absorption and 40 colorimetric analyses can be performed per day.

CHROMIUM TOTAL	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	400 mL	400 mL	25 mL	50 mL	100 mL	10 mL	2 grams	2 grams	50 grams	200 mL	1 filter strip	
Preferred Sampling Container	Plastic bottle Acid washed	Plastic bottle Acid washed	Plastic bottle Acid washed	Glass bottle	Glass bottle	Glass bottle	Pomade jar	Plastic bag	Plastic bag	Sampling jar	Envelope	
Preservation Technique	HNO ₃ pH <2	HNO ₃ pH <2	HNO ₃ pH <2	HNO ₃ pH <2	HNO ₃ pH <2	None	None	4°C	Freeze	None	None	
Operating Range	0.002 to 0.25	0.002 to 0.25	0.0005 to 0.05	0.02 to 300	0.02 to 2.5	0.25 to 300	3 to 1000	2.0 to 100	0.250 to 30	0.13 to 2.0	0.0002 to 3.5	
Limit Detection Criteria	0.002	0.002	0.0005	0.02	0.02	0.25	3	2.0	0.25	0.13	0.0002	
Std. Dev. for in run Duplicates	<20% 20-50% 50-100%											
Precision	0.04 at 0.25	0.04 at 0.25	0.0005 at 0.0046	*	0.03 at 0.50	*	4 at 38	4 at 70	-	N/A	*	
Units	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	µg/g	µg/g	µg/g	µg/m ³ 30 days	µg/in ³	

* = ± 10% at 10x Detection Limit

CHROMIUM HEXAVALENT	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	500 mL	500 mL			500 mL							
Preferred Sampling Container	Plastic bottle	Plastic bottle			Plastic bottle							
Preservation Technique	None	None			None							
Operating Range	0.01 to 1.0	0.01 to 1.0			0.01 to 0.20							
Limit Detection Criteria	0.01	0.01			0.01							
Std. Dev. for in run Duplicates	<20% 20-50% 50-100%											
Precision	0.05 at 1.0	0.05 at 1.0			0.05 at 1.0							
Units	mg/L	mg/L			mg/L							

COBALT

Cobalt is found in nature in the minerals cobaltite, smaltite, and erythrite. Weathering of igneous and sedimentary rocks and soil leaching release cobalt to the water. Cobalt is also widely used in the manufacture of alloys and pigments used in glass staining. Recently it has found use as a binder in the tungsten carbide tool industry where it is commonly added to steel as a means of improving the cutting quality of certain tools. Cobalt is also used in the electroplating industry because of its appearance, hardness and resistance to oxidation. Levels in natural waters are generally lower than 0.0001 mg/L.

Cobalt is an essential element at trace levels for both animal and plant nutrition. It is known to be one of the main constituents of Vitamin B₁₂ and its presence is believed to be instrumental in the natural synthesis of this vitamin. Cobalt deficiency in man and animals results in a type of anaemia which may be corrected by administering small dosages of cobalt chloride orally. Adverse effects due to cobalt are very slight even at high concentrations. Nausea and diarrhea are the common symptoms. No limits have been set on the maximum acceptable concentrations for cobalt in domestic water supplies. Tentative limits suggested by the Water Quality Criteria are 0.05 mg/L for continuous irrigation of all soils and 5 mg/L for use on fine textured soils. Cobalt is extremely toxic to Daphnia. The 48 hour LC 50 is 1.32 mg/L. Growth inhibition in carp has been observed above 5 mg/L.

Sampling

Special acid washed plastic containers are required for trace level determinations in domestic water and surface water samples. Glass bottles are required for domestic waste and industrial waste samples. Add 20 drops of HNO₃ per liter. Pomade jars are used for soil and sediment samples. Vegetation samples should be collected in plastic bags and refrigerated.

Method

Samples are digested with the appropriate acid solution and analyzed by atomic absorption.

Time of Analysis

Approximately 50 cobalt analyses may be performed per day.

COBALT	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	400 mL	400 mL		50 mL	100 mL		2 grams	2 grams	50 grams	200 mL		
Preferred Sampling Container	Plastic bottle Acid washed	Plastic bottle Acid washed		Glass bottle	Glass bottle		Pomade jar	Plastic bag	Plastic bag	Sampling jar		
Preservation Technique	HNO ₃ pH <2	HNO ₃ pH <2		HNO ₃ pH <2	HNO ₃ pH <2		None	4 °C	Freeze	None		
Operating Range	0.002 to 0.25	0.002 to 0.25		0.04 to 5.0	0.02 to 2.5		0.3 to 1000	1.0 to 100	0.25 to 30	0.06 to 2.0		
Limit Detection Criteria	0.002	0.002		0.04	0.02		0.3	1.0	0.25	0.06		
Std. Dev. for in run Duplicates												
Precision	0.03 at 0.25	0.003 at 0.25		0.03 at 0.25	0.04 at 0.50		0.6 at 16	4 at 50	-	-		
Units	mg/L	mg/L		mg/L	mg/L		µg/g	µg/g	µg/g	µg/m ² /30 days		

Std. Dev. for in run Duplicates

COLOR - APPARENT

Color in natural surface waters is primarily associated with the presence of humic acids derived from the decomposition of plant material such as plankton, aquatic macrophytes, terrestrial leaves and logs. Lakes containing these colored substances are commonly referred to as "acid-bog" or "brown-water" lakes. In ground waters, the color is more likely due to the presence of iron and manganese. In either case, the color is characteristically manifested as varying hues of yellowish-brown.

Water with colors of unusual hues, such as those of industrial origin, may differ greatly from the typical yellowish-brown color of natural water. Special samples known to contain organic dyes, colored metallic compounds and other types of industrial effluents cannot be adequately assessed using this particular analytical technique.

Only the "apparent color" is reported since it is considered most representative of field conditions. "Apparent color" includes the color due to dissolved substances as well as the additional color contributed by suspended matter. "True color" only considers dissolved colored substances.

Waters colored naturally by humic substances are considered unacceptable for drinking purposes because of their appearance. If the color is due to the presence of iron and manganese, it can also be associated with taste and staining problems. Most naturally colored waters are harmless; hence the specified objective of 5 Hazen Units for domestic supplies is based on aesthetic considerations rather than on known health standards.

Sampling

Samples should be collected in 1 L glass bottles.

The color of a water is greatly dependent on changes in pH, the extent of biological activity, and the precipitation of iron and manganese. To minimize changes during transit, sample containers should be filled to the neck, sealed tightly and refrigerated. Immediate delivery to the laboratory is essential.

Method

For each sample, two absorbance values are measured using a Klett-Summerson colorimeter with two filters. The apparent color is then calculated from these two values using an empirically derived equation.

Results are reported in Hazen Units.

Time of Analysis

Approximately 100 color analyses can be performed per day.

COLOUR APPARENT	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Vegh	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	75 mL	75 mL			75 mL							
Preferred Sampling Container	Glass bottle	Glass bottle			Glass bottle							
Preservation Technique	4 °C	4 °C			4 °C							
Operating Range	0 to 70	0 to 70			0 to 70							

Limit Detection Criteria

Std. Dev. for in run Duplicates
 <20%
 20-50%
 50-100%

Precision

Units

H.U.

H.U.

H.U.

H.U. = Hazen Units

CONDUCTIVITY

Conductivity is defined as the reciprocal of a water's electrical resistance measured between two electrodes one square centimeter in area and one centimeter apart. In other words, conductivity is a measure of the ability of a water to carry an electric current. The conductivity depends on the concentrations of the various ions in solutions, the mobility of each of these ions and the temperature at which the measurement is made. All results are reported relative to a standard temperature of 25°C.

Any aqueous system containing ionized substances will conduct an electric current. Most inorganic acids, bases and salts, such as hydrochloric acid, sodium bicarbonate and sodium chloride, are good conductors, while many organic compounds, such as glucose or benzene, undergo minimal dissociation and are poor conductors.

This test is so precise and accurate that it is often preferred to a dissolved solids test as an indicator of the dissolved solids content of a natural water sample. The conductivity may be correlated to the dissolved solids content by multiplying the conductivity by an appropriate factor. Generally, natural waters in Southern Ontario free of industrial wastes, have a factor of $0.65 \pm .10$. This factor may vary depending on the location.

Sampling

Aqueous samples should be collected in glass or polystyrene bottles. Soil samples should be collected in glass jars.

Care must be taken to avoid contact with any material which can release or absorb ions. Samples should not be frozen as some carbonates are precipitated and are not easily redissolved.

Method

Measurements are made using a conductivity meter. A water bath is used to adjust the sample's temperature to 25°C before the measurement is taken. Measurements can also be made on soil - water pastes, interstitial waters and leachates of soil, sediments and industrial wastes. Air bubbles and foreign material in the conductivity cell may lead to low results. Results are reported as micromhos/cm.

Time of Analysis

Approximately 200 conductivity analyses can be performed per day on aqueous samples.

CONDUCTIVITY	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	75 mL	75 mL	75 mL	75 mL	75 mL	75 mL	50 grams	grams	grams	mL		
Preferred Sampling Container	Glass bottle or polystyrene	Glass bottle	Polystyrene bottle	Glass bottle	Glass bottle	Glass bottle	Pomade jar					
Preservation Technique	None	None	None	None	None	None	None					
Operating Range	< 500	< 2000	< 150	< 2000	< 2000	*	< 2000					
Limit Detection Criteria	0.30**	0.30**	0.30**	0.30**	0.30**		-					
Std. Dev. for in run Duplicates	0.48 1.15 1.06	1.66 2.07 6.85	0.185 - -	1.70 2.34 4.29	1.70 2.34 4.29	1.70 2.34 4.29	- - -					
Precision												
Units	$\mu\text{mho/cm}$	$\mu\text{mho/cm}$	$\mu\text{mho/cm}$	$\mu\text{mho/cm}$	$\mu\text{mho/cm}$	$\mu\text{mho/cm}$	$\mu\text{mho/cm}$					

* = Test available upon request

** Based on duplicates of distilled water.

COPPER

Copper occurs naturally as the free native metal as well as in malachite, cuprite, chalcopyrite and various mixtures of sulphide ores. The presence of copper in natural waters is generally due to industrial activity, but in certain circumstances, copper salts may be introduced to a water distribution system or reservoir as a means of controlling objectionable algae slimes. Copper may also be introduced into the environment through contact with brass and copper plumbing, discharges from industries making textiles, paints and electrical products, and discharges from smelting operations and tailings ponds.

Total copper in water may occur as insoluble particulates, soluble complexes and soluble divalent ions. Copper concentrations in the Great Lakes are dependent on local inputs, but in open waters they seldom exceed 5 $\mu\text{g/L}$.

Copper is an essential element to the human body, acting as a catalyst in many biochemical reactions. It is generally agreed that copper is an active agent in the synthesis of hemoglobin and that a copper deficiency in animals causes a severe anemia similar to iron deficiency anemia. A daily intake of about 2 mg is required for normal body functions. Excessive amounts of copper are toxic, and cause abdominal pain, vomiting and convulsions. Prolonged exposure results in liver and kidney damage. Copper poisoning occurs more often in grazing livestock than in man, usually resulting from consumption of dry feeds and grasses with high copper concentrations. The free aquated copper ion is extremely toxic to fish. When bound by organic ligands, the metal is biologically less active. Copper imparts a disagreeable taste to water at concentrations well below those which would be hazardous to health; hence the drinking water standard of 1.0 mg/L is based on taste rather than on toxicity. Levels in unfiltered samples should not exceed 0.005 mg/L to protect aquatic life.

Sampling

Special acid washed plastic containers are required for trace level determinations in domestic water and surface water samples. Glass containers are required for domestic waste and industrial waste samples. Add 20 drops of HNO_3 per liter. Pomade jars are used for soil and sediment samples. Vegetation samples should be collected in plastic bags and refrigerated.

Method

Samples are digested in the appropriate acid medium and then analyzed. Sewage, sludge and Hi-Vol filters are analyzed by ICP atomic emission. All other matrices are analyzed by atomic absorption. Polarographic procedures may be used to differentiate "free" and "combined" copper.

Time of Analysis

Approximately 50 analyses can be performed per day by atomic absorption.

COPPER	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	400 mL	400 mL	25 mL	50 mL	100 mL	10 mL	2 grams	2 grams	50 grams	200 mL	1 filter strip	
Preferred Sampling Container	Plastic bottle Acid washed	Plastic bottle Acid washed	Plastic bottle Acid washed	Glass bottle	Glass bottle	Glass bottle	Pomade jar	Plastic bag	Plastic bag	Sampling jar	Envelope	
Preservation Technique	HNO ₃ pH <2	HNO ₃ pH <2	HNO ₃ pH <2	HNO ₃ pH <2	HNO ₃ pH <2	None	None	4°C	Freeze	None	None	
Operating Range	0.001 to 0.25	0.001 to 0.25	0.001 to 0.200	0.05 to 70	0.01 to 2.5	0.25 to 350	1 to 500	0.5 to 200	0.125 to 30	0.03 to 25	0.0002 to 3.5	
Limit Detection Criteria	0.001	0.001	0.001	0.05	0.01	0.25	1	0.5	0.125	0.03	0.0002	
Std. Dev. for in run Duplicates	<20%	<20%	<20%	<20%	<20%	<20%	<20%	<20%	<20%	<20%	<20%	
Precision	0.01 at 0.20	0.01 at 0.20	0.0005 at 0.013	*	0.02 at 0.40	*	0.7 at 66	0.5 at 10	-	-	*	
Units	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	µg/g	µg/g	µg/g	µg/m ² /30 days	µg/m ³	

* = ± 10% at 10x Detection Limited.

CYANIDE

Cyanides are a class of chemical compounds which contain a carbon-nitrogen constituent commonly referred to as the cyanide group. Cyanides vary in their degree of chemical activity depending on how readily they dissociate in water to form the cyanide ion. Common forms of cyanide are metal cyanide complexes, hydrocyanic acid and free cyanide ion from dissociated cyanide salts. Simple cyanides of the alkali metals readily form this ion while many metal cyanides do so at a much slower rate or not at all. The form of cyanide is also strongly dependent upon the pH. Most of the free cyanide exists as HCN at the pH values of natural waters. This form also is the most common at low pH values.

The presence of cyanide in natural waters is the result of an industrial discharge from gas works, coke ovens, gas scrubbing in steel plants and electroplating industries. Waters containing this substance are extremely toxic to virtually all aquatic life forms and are unsafe for human consumption. Cyanide exerts its toxic effects by inhibiting the normal oxidation processes in body tissue thus causing asphyxiation and death. Chlorination processes can reduce cyanide levels in raw drinking water supplies. The maximum acceptable cyanide concentration for domestic water supplies in Ontario has been set at 0.01 mg/L of cyanide as HCN. Concentrations of free cyanide in an unfiltered sample should not exceed 0.005 mg/L to protect aquatic life.

Sampling

A sample suspected of containing cyanide should never be acidified, as this will release highly toxic cyanogen gas. Samples may be preserved with 1 mL of 50% NaOH per bottle. Keep samples refrigerated and in darkness. Glass containers may be used for all types of samples, however, plastic bags are preferred for vegetation samples.

Method

Aqueous samples are distilled in-line from a weakly acidic tartaric acid solution. Analysis for "total cyanide" is then performed colorimetrically by AutoAnalyzer using chloramine-T and a barbituric acid-pyridine reagent. Soluble cyanide in soils and sediments can be determined after extraction with distilled water.

Time of Analysis

Approximately 75 cyanide analyses can be performed per day.

CYANIDE

Minimum Sample Size

Preferred Sampling Container

Preservation Technique

Operating Range

Limit Detection Criteria

Std. Dev. for in run Duplicates

Precision

Units

Surface Water

Drinking Water

Precip.

Sewage

Ind. Wastes

Sludge

Soil & Sediment

Vegⁿ

Bio Material

Dust Fall

Hi-Vol

Ambient Air

500 mL

500 mL

500 mL

500 mL

10 mL

10 grams

Glass or plastic bottle

Glass or plastic bottle

Glass bottle

Glass bottle

Glass bottle

Glass bottle

Pomade jar

Plastic bag

NaOH pH $\approx$ 10 4°C

NaOH pH $\approx$ 10 4°C

NaOH pH $\approx$ 10 4°C

NaOH pH $\approx$ 10 4°C

NaOH pH $\approx$ 10 4°C

NaOH pH $\approx$ 10 4°C

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DUSTFALL

Air pollutants in the form of dust can be a nuisance factor, since pollutants are readily perceived by the sensory organs and in some cases, the substances present in the dust may cause injury. By far the highest contributor to total dustfall is human activity. Wind occasionally contributes significant amounts.

Dust damages clothing and buildings, contaminates residential areas, damages agricultural crops and may cause eye injuries.

When assessing damages or injuries attributable to dust, an analysis of the elemental composition and a microscopic examination assist in targeting the possible toxic agents and their sources.

The ability of a dust particle to settle is mainly determined by its weight, size and surface area, wind and precipitation. The type of target also has an effect. Thus, dustfall data are affected by these external conditions. Results are used to express the atmospheric fallout of dust in specific areas.

Sampling

Samples should be analyzed within one week of collection. The American Society for the Testing of Materials (ASTM) recommends addition of water to the dustfall jar prior to installation. (Ethylene glycol may be used to prevent freezing of water in the container. Copper sulphate (15 mg/L) or mercuric chloride (0.1 g) may be used as an algicide if absolutely needed.)

Method

The method closely follows the ASTM method for the collection and analysis of dustfall. The sample is filtered to obtain soluble and insoluble fractions. The fractions are dried and weighed and the calculations performed. Extraneous materials, such as insects, foliage, algae and fungi may interfere. Results are reported as $\text{g/m}^2/30$ days.

Time of Analysis

Samples are run in batches of 36 and require 40 minutes per analysis.

DUSTFALL	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size										30 days exposure		
Preferred Sampling Container										Sampling jar		
Preservation Technique										See text		
Operating Range										>1.0		
Limit Detection Criteria										1.0		
Std. Dev. for in run Duplicates												
Precision										-		
Units										g/m ² /30 days		

FLUORIDATION RATE

Fluoride damage to vegetation is usually the result of airborne fluoride emissions which may cause chronic damage depending on the length and concentration of fumigation. The primary aluminum industry is the major source of atmospheric fluoride emissions, however, phosphate fertiliser production, elemental phosphorus plants and the primary iron, steel, and brick making industries are also significant contributors. Since the majority of fluoride emissions are in a gaseous state, such as hydrogen fluoride and silicon tetrafluoride, the fluoridation rate of the atmosphere is monitored.

Hydrogen fluoride gas severely irritates the eyes and respiratory system and may cause burns to the eyes. It may also cause irritation to the skin. The liquid causes severe, painful burns on contact with all body tissues.

The method for determining the fluoridation rate provides a relatively inexpensive surveillance method for monitoring the degree of pollution from gaseous fluorides in specified areas.

The criteria set for fluoridation rates are $40 \mu\text{g F}/100 \text{ cm}^2/30$ days for the summer growing season from April 15th to October 15th and $80 \mu\text{g F}/100 \text{ cm}^2/30$ days from October 16th to April 14th. Since the candle is exposed for 30 days, it is not possible to determine specific days of particularly high or low fluoride air pollution.

Sampling

Prior to and after exposure, the candle must be stored in a sealed, tubular container.

Method

The fluoride is fixed during exposure of the candle by reaction with Calcium oxide. The fluoride is extracted from the exposed filter paper in a buffer solution and is determined using a fluoride ion specific electrode in conjunction with a standard single junction, sleeve-type reference electrode.

Impacted fluoride particulates can lead to high results. Static monitors are subject to changes in wind speed and direction, temperature and humidity. Results are reported as $\mu\text{g F}/100 \text{ cm}^2/30$ days.

Time of Analysis

One week is required to analyze each batch of candles. Approximately 27 candles can be prepared per day.

NOTE: This test is now performed in Thunder Bay only. All samples should be sent to the Thunder Bay laboratory.

FLUORIDATION RATE

Surface Water Drinking Water Precip. Sewage Ind. Wastes Sludge Soil & Sediment Veg'n Bio Material Hi-Vol Ambient Air

Minimum Sample Size

30 days exposure

Preferred Sampling Container

Calcium oxide candle

Preservation Technique

Keep sealed when not in use

Operating Range

2 to 1000

Limit Detection Criteria

2

Std. Dev. for in run Duplicates

<20%
20-50%
50-100%

Precision

0.34 at 20

Units

$\mu\text{g}/100\text{ cm}^2$
30 days

FLUORIDE

Fluoride occurs naturally in the ionic form in natural waters, and in soils and sediments primarily as the minerals fluor spar, fluoroapatite, fluorite and cryolite. Most surface waters in Ontario contain fluoride in concentrations less than 2 mg/L although higher levels may be found in localized areas containing fluoride bearing rocks. Ground water levels may be slightly higher than surface water levels.

While weathering processes supply a large portion of the fluoride in natural waters the presence of fluoride is mainly due to the fluoridation of domestic water supplies. Fluoride concentrations near 1 mg/L are beneficial in retarding tooth decay. When fluoride is naturally present in drinking water, the concentration should not average more than 1.2 mg/L. Presence of fluoride in concentrations greater than 2.4 mg/L shall constitute grounds for rejection of the water supply. When fluoridation is practised, the permissible operating range is 0.8 mg/L to 1.2 mg/L. Fluoride concentrations between 2.4 and 10.0 mg/L can produce mottling of teeth and levels more than 50 mg/L may be toxic. Livestock drinking water should not exceed 2.0 mg/L and irrigation waters should contain less than 1.0 mg/L and 15 mg/L for use on acidic and neutral to alkaline soils respectively.

Fluoride is used in industry in a variety of manufacturing processes such as adhesives and metallurgical fluxes.

Sampling

Aqueous samples should be collected in glass or plastic containers and soil samples in pomade jars. Vegetation samples should be collected in perforated plastic bags and refrigerated.

Method

Water samples are distilled from sulphuric acid and analyzed colorimetrically on an AutoAnalyzer system by reaction with lanthanum nitrate and Alizarin Blue to form a ternary Alizarin Blue-lanthanide-fluoride complex. One laboratory uses a specific ion electrode. Vegetation samples are analyzed colorimetrically after extensive preparation. Dustfall and Hi-Vol samples are analyzed with an ion specific electrode.

Time of Analysis

Approximately 100 aqueous fluoride samples can be performed per day.

FLUORIDE	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	50 mL	50 mL	50 mL	50 mL	50 mL	2 mL	0.2 grams	1.0 grams		100 mL	2 filter strips	
Preferred Sampling Container	Glass bottle	Glass bottle	Polystyrene bottle	Glass bottle	Glass bottle	Glass bottle	Pomade jar	Plastic bag		Sampling jar	Envelope	
Preservation Technique	None	None	None	None	None	None	None	4°C		None	None	
Operating Range	0.01 to 2.0	0.01 to 2.0	0.014 to 0.250	0.01 to 2.0	0.01 to 2.0	0.05 to 4.0	12.5 to 1000	2.5 to 200		0.05 to 100	0.05 to 100	
Limit Detection Criteria	0.030	0.030	0.029	0.030	0.030	0.05	12.5	2.5		0.05	0.05	
Std. Dev. for in run Duplicates	0.009 0.013 0.018	0.009 0.013 0.018	0.0087 - -	0.009 0.013 0.018	0.009 0.013 0.018							
Precision						0.05 at 0.5	15 at 500	3 at 100		0.02 at 0.4	0.02 at 0.4	μg/m ³
Units	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	μg/g	μg/g		μg/m ² /30 days		

GLYCOLS

Glycols are alcohols having an aliphatic carbon chain and 2 hydroxy groups with the general formula $C_nH_{2n}(OH)_2$.

Ethylene glycol is used mainly for automobile antifreeze formulations and de-icing solutions. It is used as a high temperature coolant for internal combustion engines as well as in brake fluid, paints and varnishes.

Propylene glycol is also used in antifreeze and de-icing solutions and is used extensively in the food, cosmetic and pharmaceutical industries where moisture retention is important as well as in the plastic industry.

Ethylene glycol can be toxic if taken internally. Other glycols measured include 1,2-propanediol, 1,3-butanediol, 2,3-butanediol, diethylene glycol and many others.

Sampling

Aqueous samples should be collected in 1 liter glass bottles with foil lined caps. Samples should be kept cool and transported to the laboratory as quickly as possible.

Method

Aqueous samples are generally analyzed by gas chromatography following direct injection.

GLYCOLS	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	1000 mL	1000 mL		1000 mL	1000 mL	grams	grams	grams	grams			
Preferred Sampling Container	Glass bottle	Glass bottle		Glass bottle	Glass bottle							
Preservation Technique	4°C	4°C		4°C	4°C							
Operating Range	>100	>100		>100	>100							
Limit Detection Criteria	100	100		100	100							
Std. Dev. for in run Duplicates												
Precision	*	*		*	*							
Units	mg/L	mg/L		mg/L	mg/L							

* = Replicate analyses should be within 10% of the mean.

GOLD

Gold is found most often in the metallic state as native gold but is also found with tellurides. It is widely distributed and is generally found with quartz or pyrite.

Gold is best known for its use as a monetary standard and in jewelry, decoration, dental work and plating. Gold is often used as an anticorrosive as a plating on other metals. Gold alloys are used in electronic apparatus. Chlorauric acid is used in photography for toning the silver image. The Au^{198} isotope is used for treating cancer and other diseases.

Sampling

Special acid washed plastic containers are required for domestic water and surface water samples. Glass containers are required for domestic waste and industrial waste samples. Add 20 drops of HNO_3 per liter. Soil samples should be collected in pomade jars. Vegetation samples should be placed in plastic bags and refrigerated. Fish samples should be placed in plastic bags and frozen.

Method

The sample is digested with the appropriate acid mixture and analyzed by atomic absorption.

Time of Analysis

Approximately 50 gold analyses can be performed per day.

GOLD	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'h	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	400 mL	400 mL		50 mL	100 mL	10 mL	2 grams	2 grams	50 grams			
Preferred Sampling Container	Plastic bottle Acid washed	Plastic bottle Acid washed		Glass bottle	Glass bottle	Glass bottle	Pomade Jar	Plastic bag	Plastic bag			
Preservation Technique	HNO ₃ pH <2	HNO ₃ pH <2		HNO ₃ pH <2	HNO ₃ pH <2	None	None	4 °C	Freeze			
Operating Range	0.003 to 0.5	0.003 to 0.5		0.05 to 1.0	0.03 to 1.0	0.25 to 1.0	*	*	*			
Limit Detection Criteria	0.003	0.003		0.05	0.03	0.25						
Std. Dev. for in run Duplicates												
Precision	-	-		-	-	-						
Units	mg/L	mg/L		mg/L	mg/L	mg/L	µg/g	µg/g	µg/g			

* = Test available upon request.

HARDNESS

Water hardness is due mainly to the presence of calcium and magnesium salts in solution however aluminum, iron, manganese, strontium, zinc and hydrogen ions may contribute if present at high enough concentrations.

Hardness is reported as mg/L of calcium carbonate. Thus, the measured hardness is equal to the hardness which would be produced by the quoted concentration of calcium carbonate. The fraction of the total hardness accounted for by carbonate and bicarbonate salts in solution is called carbonate hardness and any excess is called noncarbonate hardness.

The hardness of waters varies widely throughout Ontario from Precambrian Shield levels of 40 - 75 mg/L, Lake Erie levels of about 140 mg/L to groundwaters which generally exceed 300 mg/L and may reach 2,000 mg/L.

Hard waters are objectionable because they form insoluble compounds with soap which greatly reduce the efficiency of the soap. At high temperatures they also form a lime scale in plumbing fixtures which can reduce the lifetimes of hot water pipes, boilers, heat exchangers, washing machines and any other appliances which use hot water.

Waters with levels above 500 mg/L are undesirable for both domestic and industrial uses.

Sampling

Sampling containers must be filled completely so that no air bubbles remain once they are capped. This precaution will prevent the loss of dissolved carbon dioxide which is essential for keeping calcium carbonate in solution. Samples should not be refrigerated as this will cause precipitation of calcium carbonate.

Method

Domestic water samples are buffered to a pH of 10 and titrated with ethylenediaminetetraacetic-acid to the Eriochrome Black T endpoint. Heavy metals may interfere by masking the endpoint, but suppression techniques alleviate this problem. Hardness levels on surface water samples are calculated from calcium and magnesium results obtained by atomic absorption.

Time of Analysis

Approximately 80 titrations can be performed per day.

HARDNESS	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	75 mL	75 mL		75 mL	75 mL							
Preferred Sampling Container	Glass bottle	Glass bottle		Glass bottle	Glass bottle							
Preservation Technique	See text	See text		See text	See text							
Operating Range		1.4 to 300		1.4 to 300	1.4 to 300							
Limit Detection		2.70		2.70	2.70							
Criteria		1.35		1.35	1.35							
Std. Dev. for in run Duplicates		0.82 0.74 0.90		0.82 0.74 0.90	0.82 0.74 0.90							
Precision												
Units	mg/L CaCO ₃	mg/L CaCO ₃		mg/L CaCO ₃	mg/L CaCO ₃							

HYDROCARBON GASES

Generally all saturated aliphatic hydrocarbon gases can be measured as well as many unsaturated ones. Methane, ethane, propanes, butanes, pentanes, ethylene, propylene, acetylene and butadiene are examples of compounds detected with the method indicated.

Methane, or marsh gas, is the major component of natural gas. Methane is used in the manufacture of several organic compounds. Air-methane mixtures are strictly monitored in coal mines because of their tendency to explode. Methane may be an asphyxiant. Methane levels in ground water should not exceed 50 cubic feet per 100,000 gallons.

Ethane is a minor component of natural gas. It is used as a refrigerant and in some fuel gases. It can be an asphyxiant.

Propane is a constituent of natural gas and crude petroleum. It is used as a fuel gas. It may be narcotic at high levels.

Butanes together with propane compose liquified petroleum gas which is used as an industrial and home heating fuel.

Pentanes are used mainly in gasoline. Trimethyl pentane is used to improve the anti-knock ratio.

Acetylene is used mainly in welding, and for cutting and soldering metals. It can be an asphyxiant and a narcotic.

Ethylene is used in welding and for cutting metals and in many synthetic reactions. Propylene is used in many synthetic reactions. Both of these gases can be asphyxiants and narcotics at high levels.

Sampling

Aqueous samples should be collected in 1 liter glass bottles with foil lined caps. Solid samples should be collected in pomade jars. These samples should be kept cool and transported to the laboratory as quickly as possible. Air samples should be collected in polyester protected aluminized sampling bags which should be stored at room temperature and in a dark place.

Method

Aqueous samples may be analyzed by gas chromatography using direct aqueous injection or by head space analysis of the vapours in a sealed bottle. Solid samples are analyzed using head space analysis of the vapours in sealed bottles fitted with "Teflon" septa. Air samples are analyzed using direct injection (sampling bags) or after carbon disulphide elution (charcoal traps).

HYDROCARBON GASES	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	1000 mL	1000 mL		1000 mL	1000 mL	20 grams	20 grams	20 grams	20 grams			½ full bag
Preferred Sampling Container	Glass bottle	Glass bottle		Glass bottle	Glass bottle	Glass bottle	Pomade jar	Pomade jar	Pomade jar			Sampling bag
Preservation Technique	4 °C	4 °C		4 °C	4 °C	4 °C	4 °C	4 °C	4 °C			Keep in dark place
Operating Range	>10	>10		>10	>10	>1.0	>1.0	>1.0	>1.0			>100
Limit Detection Criteria	10	10		10	10	1.0	1.0	1.0	1.0			100
Std. Dev. for in run Duplicates												
Precision	*	*		*	*	*	*	*	*			*
Units	µg/L	µg/L		µg/L	µg/L	µg/g	µg/g	µg/g	µg/g			µg/m³

* = Replicate analyses should be within 10% of the mean.

IRON (TOTAL)

Iron is the most abundant of the heavy metals in nature, but is generally found at low levels in most surface waters because of the relatively low solubility of ferric iron when the pH exceeds 7. Compounds of iron in water are in a state of dynamic chemical equilibrium caused by bacterial activity and chemical oxidation. Under reducing conditions, iron exists in the ferrous state. On exposure to the atmosphere or oxidants, ferrous iron is oxidized to the ferric state and may hydrolyze to form insoluble ferric oxides. Because the relative concentrations of these compounds are constantly changing, only a total iron determination provides relevant data. The most common forms of iron in water include ferrous ions, ferric ions, inorganic and organic chelates, complex colloidal organics, suspended oxides and hydroxides. Iron levels in surface waters are generally less than 0.5 mg/L but they often exceed this in groundwaters. Iron is added to the environment by weathering of iron bearing minerals such as pyrite, hematite and magnetite, mineral processing and acid mine drainage.

The role of iron is important in eutrophication studies as it affects retention and release mechanisms of phosphorus components in lake and river sediments. This ability of iron to bind up phosphorus is also important in nutrient removal programs at water pollution control plants where ferric chloride is sometimes used as a phosphate precipitant.

Iron is an important component of hemoglobin and an essential element for all life forms. It is non-toxic at high levels but is objectionable in domestic supplies because of the color and bitter taste it imparts. It is also known to cause reddish-brown stains on plumbing fixtures and laundry. The water quality objective for iron in domestic water supplies is 0.3 mg/L (filterable). Levels in an unfiltered sample should not exceed 0.3 mg/L to protect aquatic life.

Sampling

Only total iron analyses are performed routinely in water samples. If filterable iron is requested, samplers must request a total iron analysis on a field filtered sample. For trace level analysis use a special acid washed plastic bottle and add 20 drops of HNO_3 per liter. Glass containers should be used for sewage, industrial waste, sludge, soil and sediment samples. Vegetation samples should be collected in plastic bags and refrigerated.

Method

River and drinking water samples are digested and analyzed colorimetrically by an automated TPTZ method. Other samples are digested with an appropriate acid mixture and analyzed. Sewage, sludge and Hi-Vol samples are analyzed by ICP atomic emission. All other samples are analyzed by atomic absorption.

Time of Analysis

Approximately 150 iron analyses can be performed per day by the colorimetric method and 50 per day at atomic absorption.

IRON TOTAL	Surface Water	Drinking Water	Precip. Sewage	Ind. Wastes	Sludge	Soil & Sediment	Vegh	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	100 mL	100 mL	25 mL	50 mL	10 mL	2 grams	2 grams	50 grams	200 mL	1 filter strip	
Preferred Sampling Container	Glass or polystyrene	Glass bottle	Plastic bottle Acid washed	Glass bottle	Glass bottle	Pomade jar	Plastic bag	Plastic bag	Sampling jar	Envelope	
Preservation Technique	None	None	HNO ₃ pH <2	HNO ₃ pH <2	None	None	4 °C	Freeze	None	None	
Operating Range	0.02 to 1.00	0.03 to 2.00	0.001 to .200	0.20 to 300	1.00 to 1500	10 to 2500	10 to 1000	2.5 to 125	0.06 to 50	0.001 to 1.5	
Limit Detection Criteria	0.046	0.046	0.001	0.20	1.0	10	10	2.5	0.06	0.001	
Std. Dev. for in run Duplicates	0.014 0.078 0.025	0.021 0.040 0.039	0.004 at 0.132	*	0.12 at 2.00	7,900 at 56,000	7 at 100	-	-	*	
Precision	0.05 at 1.00	0.050 at 1.00	0.004 at 0.132	mg/L	mg/L	μg/g	μg/g	μg/g	μg/m ² / 30 days	μg/m ³	
Units	mg/L	mg/L	mg/L	mg/L	mg/L	μg/g	μg/g	μg/g	μg/m ² / 30 days	μg/m ³	

* = ± 10% at 10x Detection Limit

LEAD

Lead is found in nature primarily as the mineral galena (lead sulphide) but also as cerussite, anglesite, crocoite, wulfenite, pyromorphite, matlockite and vanadate. Since the inorganic salts of lead are relatively insoluble, the element most commonly enters a water supply via industrial, mine and smelter discharges or through dissolution of old corroded lead plumbing. The latter problem is much more common in areas where the water is "soft"; that is, it has a low hardness or mineral content. Other sources of lead include the burning of leaded fuels and the production of lead batteries, explosives and paints. Levels in surface waters generally are below 0.04 mg/L.

Lead is an extremely toxic element which tends to exchange with calcium and accumulate in bone marrow. Lead toxicity may not appear until many years after the last exposure. Organic compounds of lead are many times more toxic than inorganic forms. The most common example is tetraethyl lead which is used as an "antiknock" additive in motor fuels. Organic lead exerts its toxic effect on the nervous system within a very short time causing mental confusion, delirium, nausea, hallucinations, insomnia, and convulsions. The maximum acceptable concentration of lead in domestic water is 0.05 mg/L. The toxicity of lead to aquatic life is highly dependent on the alkalinity. The toxicity generally decreases as the alkalinity increases. The total lead concentrations should not exceed the values below.

Alkalinity mg/L as CaCO_3	Maximum Lead Concentration $\mu\text{g/L}$
<20	5
20-40	10
40-80	20
>80	25

Sampling

When levels below the normal range of application are required, a notation must be included on the sample submission form so that the sample will be preconcentrated prior to analysis. One full 32 oz bottle is required exclusively for the preconcentration process.

Special acid washed plastic containers are required for trace level determinations in domestic water and surface water samples. Glass containers are required for domestic waste and industrial waste samples. Add 20 drops of HNO_3 per liter. Pomade jars should be used for soil and sediment samples. Vegetation samples should be collected in plastic bags and refrigerated.

Method

Samples are digested with the appropriate acid mixture and analyzed. Sewage and sludge samples are analyzed by ICP atomic emission. Hi-Vol samples are analyzed by X-ray fluorescence. All other samples are analyzed by atomic absorption. A polarographic method can also be used for low concentrations in some aqueous matrices.

Time of Analysis

Approximately 50 lead analyses can be performed per day.

LEAD	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	400 mL	400 mL	25 mL	50 mL	100 mL	10 mL	2 grams	2 grams	50 grams	200 mL	1 filter circle	
Preferred Sampling Container	Plastic bottle Acid washed	Plastic bottle Acid washed	Plastic bottle Acid washed	Glass bottle	Glass bottle	Glass bottle	Pomade jar	Plastic bag	Plastic bag	Sampling jar	Envelope	
Preservation Technique	HNO ₃ pH <2	HNO ₃ pH <2	HNO ₃ pH <2	HNO ₃ pH <2	HNO ₃ pH <2	None	None	4°C	Freeze	None	None	
Operating Range	0.003 to 0.25	0.003 to 0.25	0.001 to 0.200	0.10 to 70	0.03 to 2.5	0.10 to 70	3 to 1000	2 to 400	0.75 to 30	0.12 to 30	0.10 to 0.30	
Limit Detection Criteria	0.003	0.003	0.001	0.10	0.03	0.10	3	2	0.75	0.12	0.10	
Std. Dev. for in run Duplicates												
Precision	0.037 at 0.25	0.037 at 0.25	0.001 at 0.017	*	0.04 at 0.50	*	1.7 at 68	12 at 200	-	-	0.3 at 3	
Units	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	μg/g	μg/g	μg/g	μg/m ² /30 days	μg/m ³	

* ± 10% at 10x Detection Limit.

LITHIUM

The element lithium is comparatively rare. It is concentrated in complex silicate minerals such as petalite and spodumene. Lithium is easily weathered and because lithium compounds are very soluble, they tend to stay in solution. It is rarely found in surface waters and water supplies, but it has been detected in hot springs and highly mineralized brines where the concentration is usually less than 10 mg/L. The major source of lithium in natural waters is industrial discharge.

Lithium is used in alloys, organic syntheses and special glasses. Lithium chloride and lithium stearate are used in de-humidifiers. Lithium hydride is very corrosive and irritating.

Lithium becomes toxic to animals and humans only if taken in very large amounts. When in sufficient quantity, it has the capacity for replacing sodium in body cells and thus upsetting intra-cellular metabolism. Cause of death is generally due to kidney failure although secondary effects on the nervous system are also observed. Small doses of lithium carbonate have been effectively used as an anti-depressant for treatment of psychiatric patients. No guidelines have yet to be set for drinking waters, but irrigation water must contain less than 2.5 mg/L.

Sampling

Special acid washed containers are required for trace level determinations in domestic water and surface water samples. Glass bottles are required for domestic waste and industrial waste samples. Add 20 drops of HNO_3 per liter. Soil samples should be collected in pomade jars. Vegetation should be placed in plastic bags and refrigerated. Fish samples should be placed in plastic bags and frozen.

Method

Samples are digested with the appropriate acid mixture and analyzed by atomic absorption.

Time of Analysis

Approximately 50 lithium analyses can be performed per day.

LITHIUM	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	400 mL	400 mL	mL	400 mL	400 mL	10 mL	2 grams	2 grams	50 grams	200 mL		
Preferred Sampling Container	Plastic bottle Acid washed	Plastic bottle Acid washed		Glass bottle	Glass bottle	Glass bottle	Pomade jar	Plastic bag	Plastic bag	Sampling jar		
Preservation Technique	HNO ₃ pH <2	HNO ₃ pH <2		HNO ₃ pH <2	HNO ₃ pH <2	None	None	4 °C	Freeze	None		
Operating Range	0.0005 to 0.10	0.0005 to 0.10		*	0.005 to 1.0	*	*	*	*	*		
Limit Detection Criteria	0.0005	0.0005			0.0005							
Std. Dev. for in run Duplicates												
Precision	0.002 at 0.02	0.002 at 0.02			0.002 at 0.02							
Units	mg/L	mg/L		mg/L	mg/L	mg/L	µg/g	µg/g	mg/g	µg/m ² /30 days		

* = Test available upon request.

MAGNESIUM

Magnesium ranks eighth in the natural order of elemental abundance and is commonly found in the minerals magnesite, dolomite, olivine, serpentine, and talc and asbestos. Its industrial applications include the manufacture of metal alloys, photographic flash bulbs, flares, cement, tanning solutions and explosives. It is also used in milk of magnesia and epsom salts.

Magnesium is present in all natural waters and is generally associated with the presence of calcium. In natural fresh water systems the calcium concentration is approximately three to five times that of magnesium. If the reverse is found, an artificial source of magnesium is probable. Natural processes generally add more magnesium to the environment than do industrial processes.

Magnesium is an essential element for the life functions of all plants and animals. It is closely associated with the metabolic activity of phosphorus where it is believed to act as an activator of phosphate enzymes. Ingestion of magnesium produces no adverse effects at levels normally encountered in domestic water supplies, but concentrations in excess of 125 mg/L exert a cathartic action on the gastro-intestinal tract.

Magnesium is a major contributor to water "hardness"; therefore limitations on the amount of magnesium in water supplies are of concern especially to industry (see Hardness). Magnesium may also impart a disagreeable taste.

Sampling

Routine surface and domestic water samples should be collected in 1 L glass bottles. Precipitation samples should be collected in polystyrene bottles.

Non-routine industrial waste samples, or any other samples where high levels of heavy metals are suspected, should be sampled in acid washed plastic containers and preserved with 20 drops of nitric acid per liter. Pomade jars should be used for soil and sediment samples. Vegetation samples should be collected in plastic bags and refrigerated. Fish samples should be placed in plastic bags and frozen.

Method

Surface water and precipitation samples are analyzed directly by atomic absorption. Drinking water results are done by calculation. Other samples are digested with an appropriate acid mixture depending on the sample matrix and the laboratory section doing the work. Sewage samples are analyzed by ICP atomic emission. All other matrices are analyzed by atomic absorption.

Time of Analysis

Approximately 50 magnesium analyses can be performed per day.

MAGNESIUM	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	100 mL	100 mL	100 mL	50 mL	100 mL	10 mL	2 grams	2 grams	50 grams	200 mL	1 filter strip	
Preferred Sampling Container	Glass or polystyrene	Glass bottle	Polystyrene bottle	Glass bottle Acid washed	Glass bottle Acid washed	Glass bottle	Pomade jar	Plastic bag	Plastic bag	Sampling jar	Envelope	
Preservation Technique	None	None	None	HNO ₃ pH <2	HNO ₃ pH <2	None	None	4 °C	Freeze	None	None	
Operating Range	0.07 to 5.00	0.07 to 5.00	0.01 to 2.00	0.1 to 300	0.002 to 1.0	0.5 to 1500	1 to 500	2 to 200		0.12 to 4.0		
Limit Detection Criteria	0.138	0.138	0.013	0.1	0.002	0.5	1.0	2		0.12		
Std. Dev. for in run Duplicates	0.042 0.044 0.055											
Precision	0.003 at 0.05	0.003 at 0.05		*	0.03 at 0.5	0.50 at 500	136 at 3700	7 at 100		-		
Units	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	µg/g	µg/g	µg/g	µg/m ² / 30 days	µg/m ³	

MANGANESE

Manganese is a common element in nature and found in numerous minerals as an oxide, sulphide, carbonate and a silicate. It is found at very low levels in natural waters, generally in the divalent (soluble) or quadrivalent (suspended) state, but in the absence of dissolved oxygen, the divalent state predominates. The permanganate anion, containing manganese in the heptavalent state, is not found in water unless it has been added artificially. This test does not distinguish between the various oxidation states of manganese. High levels of manganese are usually the result of industrial discharges. Common industrial applications include the manufacture of metal alloys, dry cell batteries, paints, varnishes, inks, dyes, glass, ceramics, matches, fireworks, and fertilizer. Iron and steel plants and acid mine drainage account for a large portion of the manganese found in the environment.

Manganese in trace quantities is essential for the proper nutrition of both animals and plants and is believed to be important in the metabolic oxidation-reduction processes of living cells. Manganese deficiency in animals is characterized by lack of growth, bowing and abnormal fragility of the bones, and degeneration of the reproductive organs. Manganese and its salts, ingested in very large quantities, are potent nerve toxins.

Although manganese is non-toxic at the levels commonly encountered in water supplies, it causes unpleasant tastes, stains laundry and plumbing fixtures, and encourages the growth of objectionable micro-organisms at treatment plants. The maximum acceptable concentration of manganese for domestic water supplies in Ontario is 0.05 mg/L.

Sampling

Routine surface and domestic water samples should be collected in 1 L glass bottles.

Special acid washed plastic containers are required for trace level determinations in domestic water, precipitation and surface water samples. Glass containers are required for domestic waste and industrial waste samples. Add 20 drops of HNO_3 per liter. Pomade jars should be used for soil and sediment samples. Vegetation samples should be collected in plastic bags and refrigerated.

Method

River samples are digested in sulphuric acid-hydroxylamine media and analyzed colorimetrically with a formaldoxime method which has been adapted to an AutoAnalyzer system. Manganese analyses on other matrices are performed by ICP atomic emission or atomic absorption following digestion in an appropriate acid mixture.

Time of Analysis

Approximately 150 manganese analyses can be performed per day by the colorimetric method and 50 by atomic absorption.

MANGANESE	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Vegh	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	75 mL	75 mL	25 mL	50 mL	100 mL	10 mL	2 grams	2 grams	50 grams	200 mL	1 filter strip	
Preferred Sampling Container	Glass or polystyrene	Glass or polystyrene	Plastic bottle Acid washed	Glass bottle	Glass bottle	Glass bottle	Pomade jar	Plastic bag	Plastic bag	Sampling jar	Envelope	
Preservation Technique	None	None	HNO ₃ pH <2	HNO ₃ pH <2	HNO ₃ pH <2	None	None	4°C	Freeze	None	None	
Operating Range	0.002 to 0.20	0.002 to 0.20	0.001 to 0.200	0.05 to 70	0.02 to 2.5	0.25 to 350	1 to 500	1 to 200	1.00 to 25.0	0.01 to 1.0	0.0002 to 0.35	
Limit Detection Criteria	0.004	0.004	0.001	0.05	0.02	0.25	1.00	1.00	1.00	0.01	0.0002	
Std. Dev. for in run Duplicates	0.0012 0.0021 0.0029	0.0012 0.0021 0.0029										
Precision	0.03 at 0.20	0.03 at 0.20	0.0002 at 0.007	*	0.03 at 0.40	*	350 at 2720	4 at 60	-	-	*	
Units	mg/l	mg/L	mg/L	mg/L	mg/L	mg/L	μg/g	μg/g	μg/g	μg/m ² /30 days	μg/m ³	

* = ± 10% at 10x Detection Limit.

MATRIX ELEMENTS IN VEGETATION BY X-RAY FLUORESCENCE

Vegetation is composed mainly of carbon, hydrogen, oxygen, nitrogen and eight "matrix elements". These matrix elements are considered to be sodium, potassium, calcium, magnesium, silicon, phosphorus, sulphur and chlorine as these elements are typically found in concentrations in excess of 1000 ppm. A comparison of matrix element concentrations in damaged vegetation with those considered normal for a particular species provides a relatively rapid method of assessing possible pollution damage to vegetation.

Only potassium, calcium, silicon, phosphorus, sulphur and chlorine are measured routinely by this procedure.

Several other tests are performed on vegetation samples. For additional material, the reader is referred to the appropriate individual test outlines.

Sampling

At least 2 grams of powdered vegetation are required. Sample should be collected in perforated plastic bags, refrigerated and transported to the laboratory as quickly as possible.

Method

Samples are pelletized and analyzed by measuring the fluorescent intensity for each of the six elements for 8 seconds. A linear calibration is used to convert intensity readings to concentrations. The concentrations are corrected for inter-element effects.

Time of Analysis

Approximately 100 samples can be weighed and pelletized per day. Approximately 140 samples can be analyzed for all six elements per day.

RANGE, PRECISION, DETECTION LIMITS FOR
THE 6 MATRIX ELEMENTS IN VEGETATION

Minimum samples size
Preferred sampling container
Preservation technique

2 g dried vegetation
Perforated plastic bag
Refrigerate

Element	Range	Precision	Lower Detection Limit
Silicon	0 - 0.5% 1.0 - 2.0%	0.03%	0.03%
Phosphorus	0 - 0.2% 0.2 - 0.4% 0.4 - 0.6%	0.02%	0.02%
Sulphur	0 - 0.5% 0.5 - 1.0% 1.0 - 2.0% 2.0 - 4.0%	0.02%	0.02%
Chlorine	0 - 0.5% 0.5 - 1.0% 1.0 - 3.0%	0.01%	0.01%
Potassium	0 - 1.0% 1.0 - 2.0% 2.0 - 4.0%	0.04%	0.02%
Calcium	0 - 1.0% 1.0 - 2.5% 2.5 - 4.5%	0.07%	0.02%

MERCURY

Mercury is a relatively rare element often found as the mineral cinnabar (mercuric sulphide). Mercury is seldom present in natural waters at levels exceeding 50 parts per billion. The solubility of metallic mercury in water is low and the solubility of ionic mercury compounds depends upon the actual conditions of the solubilizing water (i.e. acidity, presence of complexing anions, other organo-metallic complexes). As a result, the mercury content of surface water depends upon the accessibility of mercury, time of contact and conditions of the solubilizing media.

Mercury has many industrial applications which include (in order of consumption) electrical apparatus, chloralkali industry, paint, industrial control instruments, agriculture, dental applications, general laboratory use, and pulp and paper production.

The greatest environmental hazard associated with mercury is its tendency to become concentrated in living organisms. Any form of mercury entering an aquatic system could be converted via bacterial activity to methyl mercury derivatives. These substances can then become concentrated along a food chain until they reach toxic levels and begin to endanger higher forms of life. In man, mercury exerts its toxic effects on the nervous system. Common symptoms include muscular tremors, speech impairment, lack of co-ordination and emotional disturbances. Concentrations of total mercury in filtered samples should not exceed 0.1 $\mu\text{g/L}$ to protect aquatic life, nor should the levels of total mercury in fish exceed 0.5 $\mu\text{g/g}$. Mercury is a substance with a zero tolerance level.

Sampling

Aqueous samples should be collected in 176 mL glass bottles, similar to the microbiology bottles. Preservation is accomplished by adding 1 mL of concentrated nitric acid (Baker analyzed only) per bottle and 0.5 mL of potassium dichromate solution (saturated) to maintain a slight yellow color. Sludge samples should be collected in 1 L glass bottles and preserved with only nitric acid. Soil and dried vegetation samples should be collected in pomade jars. Fish samples should be placed in plastic bags and frozen. Samples stored for over 3 days, even when preserved, may lose mercury. Immediate delivery to the laboratory is essential. Consultation with laboratory staff is advisable whenever mercury analyses are required.

Method

Samples are digested using an appropriate acid mixture and analyzed by cold vapour flameless atomic absorption.

Time of Analysis

Approximately 20 mercury analyses can be performed per day.

MERCURY	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	176 mL	176 mL		100 mL	100 mL	500 mL	10 grams	3 grams	50 grams			
Preferred Sampling Container	Glass Micro-biology bottle	Glass Micro-biology bottle		Glass bottle	Glass bottle	Glass bottle	Pomade jar	Pomade jar	Plastic bag			
Preservation Technique	K ₂ Cr ₂ O ₇ HNO ₃	K ₂ Cr ₂ O ₇ HNO ₃		K ₂ Cr ₂ O ₇ HNO ₃	K ₂ Cr ₂ O ₇ HNO ₃	HNO ₃	None	None	Freeze			
Operating Range	0.04 to 0.4	0.04 to 0.4		0.04 to 0.4	0.04 to 0.4	10 to 300	0.01 to 3	0.01 to 3	0.01 to 2			
Limit Detection Criteria	0.04	0.04		0.04	0.04	10	0.01	0.01	0.01			
Std. Dev. for in run Duplicates												
Precision	0.07 at 0.7	0.07 at 0.7		0.07 at 0.7	0.07 at 0.7	150 at 1000	0.09 at 1.1	0.09 at 1.1	0.12 at 2.2			
Units	µg/L	µg/L		µg/L	µg/L	µg/L	µg/g	µg/g	µg/g			

<20%
 20-50%
 50-100%

MICROBIOLOGICAL INDICATORS OF FECAL POLLUTION

Escherichia coli (E. coli) - Experimental procedure

E. coli is the predominant, facultative bacterial species in the large bowel and is thus the coliform most directly related to fecal pollution. E. coli is occasionally pathogenic to man (e.g. urinary tract infections) but its primary importance in water bacteriology is as an indicator.

Methods developed historically for the detection of fecal pollution have not been sufficiently specific for E. coli. However, a recently developed medium designed specifically for the detection of E. coli has been tested in this laboratory and proven successful. It is now possible to determine concentrations of E. coli on a limited number of samples.

This technique can only be performed with prior consultation and is not available for routine work.

Sampling

Samples should be collected in pre-sterilized glass bottles with red labels (thiosulphate added) or in rubber syringes. Samples should be packed in ice but should not be frozen.

E. coli levels will undergo change even upon refrigeration; therefore, immediate delivery to the laboratory is essential. Analyses should be performed as soon as possible after collection, preferably within six hours, but not exceeding 24 hours.

Method

Analysis for E. coli requires filtration of an appropriate volume or dilution of the sample and incubation on mTEC Agar. The plates are incubated to allow a gradual increase to $44.5 \pm 0.5^{\circ}\text{C}$ over approximately 4 hours. Total incubation time is 22 ± 1 hours. The filter is then transferred to a filter pad saturated with urease reagent and allowed to stand for 15 minutes. Urease positive colonies will turn magenta. Using a stereoscopic microscope (10X), all colonies that remain yellow, yellow-green or yellow-brown, are counted as E. coli.

Time of Analysis

The number of analyses performed per day will depend on the type of sample, the number of dilution steps required, and the incubation period. E. coli results will be available 2 days after sample submission.

FECAL POLLUTION INDICATORS
E. COLI

	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Vegh	Bio Material	Dust Fall	Hi-Vol	Ambient Air
--	---------------	----------------	---------	--------	-------------	--------	-----------------	------	--------------	-----------	--------	-------------

Minimum Sample Size	150 mL	150 mL		150 mL	*	*	* grams					*
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Preferred Sampling Container	See text	See text		See text								
------------------------------	----------	----------	--	----------	--	--	--	--	--	--	--	--

Preservation Technique	Pack in ice	Pack in ice		Pack in ice								
------------------------	-------------	-------------	--	-------------	--	--	--	--	--	--	--	--

Operating Range

Limit Detection Criteria

Std. Dev. for in run Duplicates

< 20%
20-50%
50-100%

Precision

Units

* = Available upon request.

MICROBIOLOGICAL INDICATORS OF FECAL POLLUTION

Fecal Coliforms (FC) Procedure

The FC group of bacteria is basically a subgroup of the Total coliforms and is comprised of aerobic and facultative anaerobic, Gram-negative, non-sporeforming, rod-shaped bacteria which ferment lactose at 44.5°C . The FC parameter is a more specific test for bacteria associated with human and animal fecal material. This test has proved useful as an indicator of relatively recent fecal pollution inputs, however, it is by no means completely selective for Escherichia coli, the coliform most directly related to fecal pollution. Bacteria related to other forms of input such as Klebsiella may be detected with this test.

Sampling

Samples should be collected in pre-sterilized glass bottles with red labels (thiosulphate added) or rubber syringes. Samples should be packed in ice but should not be frozen.

FC levels will undergo change even upon refrigeration; therefore, immediate delivery to the laboratory is essential. Analyses should be performed as soon as possible after collection, preferably within six hours, but not exceeding 24 hours (in some cases 3 days is allowed but this procedure is not advised). The necessary frequency of sampling is outlined in the MOE Water Management booklet (1978).

Method

Analyses for fecal coliforms are by membrane filtration. The membrane is placed on a petri dish containing mTEC agar and incubated to allow a gradual increase to $44.5 \pm 0.5^{\circ}\text{C}$ over a four hour period. The total incubation time is 22 ± 1 hours.

After incubation, the number of fecal coliform colonies are determined with a stereoscopic microscope at 10X magnification. All yellow, yellow-brown and yellow-green colonies are counted.

Time of Analysis

The number of analyses performed per day will depend on the type of sample, the number of dilution steps required, the number of parameters requested per sample and the incubation periods for each parameter. FC results are generally available within 48 hours of submission.

FECAL POLLUTION INDICATORS
FECAL COLIFORMS

	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	150 mL	150 mL		150 mL	* mL	* mL	* grams					*
Preferred Sampling Container	See text	See text		See text								
Preservation Technique	Pack in ice	Pack in ice		Pack in ice								
Operating Range												

Limit Detection Criteria

Std. Dev. for
 <20%
 20-50%
 50-100%
 in run
 Duplicates

Precision

Units

* = Available upon request

MICROBIOLOGICAL INDICATORS OF FECAL POLLUTION

Fecal Streptococci (FS) Procedure

The FS group of bacteria is comprised of aerobic, spherical to ovoid Gram positive cells occurring singly, in pairs or in short or long chains. When used as indicators of fecal contamination, the following species and varieties are implied: Streptococcus faecalis, S. faecalis var. liquefaciens, S. faecalis var. zymogenes, S. faecium, S. faecium var. durans, S. bovis and S. equinus. FS can best be used in conjunction with the FC parameter to indicate the nature of the fecal source. If the ratio of the geometric mean densities of FC to FS at pollution sources or outfalls exceeds 4, then the source is likely human in origin. A ratio of 0.7 or less suggests a non-human source. Ratios between these values are difficult to interpret and may be mixtures. Numerous environmental factors may influence the densities of FC and FS and thus care must be used in interpretation. This ratio is only valid when the FC density approached or exceeds 100/100 mL.

Sampling

Samples should be collected in pre-sterilized glass bottles with red labels (thiosulphate added) or rubber syringes. Samples should be packed in ice but should not be frozen.

FS levels will undergo change even upon refrigeration; therefore, immediate delivery to the laboratory is essential. Analyses should be performed as soon as possible after collection, preferably within six hours, but not exceeding 24 hours (in some cases 3 days is allowed but this procedure is not advisable). The necessary frequency of sampling is outlined in the MOE Water Management booklet (1978).

Method

Analysis for FS is by membrane filtration (HAMES). The membrane is placed on m-Enterococcus Agar (Difco) and incubated at $35.0 \pm 0.5^{\circ}\text{C}$ for 48 ± 3 hours. Using a stereoscopic microscope (10X), all colonies that are red, maroon or pink are counted as fecal streptococci.

Time of Analysis

The number of analyses performed per day will depend on the type of sample, the number of dilution steps required, the number of parameters requested and the incubation periods for each parameter. FS results are usually available within 72 hours of submission.

FECAL POLLUTION INDICATORS
FECAL STREPTOCOCCI

	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Vegh	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	150 mL	150 mL		150 mL	*	*	* grams					*
Preferred Sampling Container	See text	See text		See text								
Preservation Technique	Pack in ice	Pack in ice		Pack in ice								
Operating Range												

Limit Detection Criteria

Std. Dev. for in run Duplicates
<20%
20-50%
50-100%

Precision

Units

* = Available upon request

MICROBIOLOGICAL INDICATORS OF FECAL POLLUTION

Presence-Absence (P-A) Procedure

The P-A procedure has been developed as an alternative to the membrane filtration procedure and is a sensitive means of detecting pollution indicator bacteria in drinking water samples. Essentially, the P-A test is a modification of the most probable number (MPN) procedure, which uses a dilution series of broth tubes to detect and enumerate microorganisms. The P-A test employs only one analysis bottle per sample. The test allows for an inoculum of up to 100 mL and will qualitatively determine a wide variety of pollution indicator bacteria. They include total coliforms, fecal coliforms, fecal streptococci, Pseudomonas aeruginosa, Staphylococcus aureus, Aeromonas sp. and Clostridium perfringens.

Sampling

Samples should be collected in pre-sterilized bottles with red labels (thiosulphate added) or rubber syringes. Samples should be packed in ice but should not be frozen.

Bacterial levels will undergo change even upon refrigeration; therefore, immediate delivery to the laboratory is essential. Analyses should be performed as soon as possible after collection, preferably within 6 hours, but not exceeding 24 hours if feasible. Sample analysis will not be performed on samples older than 3 days. The necessary frequency of sampling is outlined in the MOE Drinking Water Objectives (1976) booklet.

Method

The P-A test involves the inoculation of 50 or 100 mL of water into a bottle of MacConkey broth (HAMES). The bottle is incubated at $35 \pm 0.5^{\circ}\text{C}$ for up to four days. If growth occurs with the production of acid and/or gas, then other tests are used to determine the types of bacteria present.

Time of Analysis

The time required for completion of test procedures associated with the P-A test is up to one week depending on the level of pollution in the sample. A sample containing a variety of indicator bacteria will require several confirmation tests before results can be reported. Drinking water samples which show the presence of fecal bacteria within 24 hours will have this information phoned to the municipality as soon as confirmed results are available.

FECAL POLLUTION INDICATORS PRESENCE-ABSENCE

	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	150 mL	150 mL		150 mL	*	* mL	* grams					*
Preferred Sampling Container	See text	See text		See text								
Preservation Technique	Pack in ice	Pack in ice		Pack in ice								
Operating Range												

Limit Detection Criteria

Std. Dev. for in run Duplicates

Precision

Units

* = Available upon request.

MICROBIOLOGICAL INDICATORS OF FECAL POLLUTION

Pseudomonas aeruginosa (*P. aeruginosa*) Procedure

P. aeruginosa is a motile, Gram-negative rod which does not ferment lactose and is oxidase positive.

Isolates of *P. aeruginosa* are found in human feces and their presence in water is generally the direct cause of recent fecal waste inputs from a nearby source. Their presence is thus a good indication of a potential health hazard.

P. aeruginosa is also an opportunistic pathogen and has been identified as the causative agent of a number of infections that may be transmitted through a contaminated water body to a susceptible host. Of chief concern to bathers is the association between *P. aeruginosa* and the disease known as otitis externa "swimmer's ear".

Sampling

Samples should be collected in pre-sterilized glass bottles with red labels (thiosulphate added) or in rubber syringes. Samples should be packed in ice but should not be frozen.

P. aeruginosa levels will undergo change even upon refrigeration; therefore, immediate delivery to the laboratory is essential. Analyses should be performed as soon as possible after collection, preferably within six hours, but not exceeding 24 hours.

Method

An appropriate volume or dilution of the sample is filtered through a 0.45 μ membrane filter and incubated in mPA Agar at $41.5 \pm 0.5^\circ\text{C}$ for 48 ± 2 hours.

Using a stereoscopic microscope (10X), all tan to dark-brown or black colonies are counted as *Pseudomonas aeruginosa*.

Time of Analysis

The number of analyses performed per day will depend on the type of sample, the number of dilution steps required, the number of parameters requested and the incubation periods for each parameter. Consultation is necessary prior to this parameter being requested. *P. aeruginosa* results are usually available 72 hours after submission.

FECAL POLLUTION INDICATORS P. AERUGINOSA

	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	150 mL	150 mL		150 mL	* mL	* mL	* grams					*
Preferred Sampling Container	See text	See text		See text								
Preservation Technique	Pack in ice	Pack in ice		Pack in ice								
Operating Range												

Limit Detection Criteria

Std.
Dev.
for
in run
Duplicates

< 20 %
20 - 50 %
50 - 100 %

Precision

Units

* = Available on request.

MICROBIOLOGICAL INDICATORS OF FECAL POLLUTION

Salmonella - Non-routine Procedure

Salmonella is one of the most important and common waterborne pathogenic organisms. The members of this genus are widely distributed in the environment and are probably responsible for most of the recognized waterborne disease outbreaks. Salmonella can be frequently detected in sewage, stormwater and polluted rivers and lakes. Fecal contamination from animals, particularly domestic, and insufficiently treated human wastes are the major source of these organisms.

Since Salmonella serotypes are all potentially pathogenic, their frequent isolation is taken as proof of a public health hazard.

The methods required are complex and slow, therefore their use is recommended only for specific studies and only with consultation and careful planning.

Sampling

Samples should be collected in pre-sterilized glass bottles with red labels (thiosulphate added). Samples should be packed in ice but should not be frozen.

Sampling can also be carried out with Moore Swabs (gauze pad) placed at appropriate locations in sampling areas.

Salmonella levels will undergo change even upon refrigeration; therefore, immediate delivery to the laboratory is essential. Analyses should be performed as soon as possible after collection, preferably within six hours, but not exceeding 24 hours.

Method

There are several procedures for the isolation and identification of Salmonella from waters and wastewaters. The method chosen will depend on the survey conducted. All the procedures include sample concentration (Moore swab or membrane filtration), enrichment in selective broths, plating on selective agar media and identification by a variety of biochemical and serological tests.

Time of Analysis

The number of samples analyzed will depend on the volume and type of sample and, more specifically, on the methods necessary for analysis. In general, a five-day period is required for Salmonella analyses.

Consultation with Microbiology staff prior to the use of this parameter is absolutely essential.

FECAL POLLUTION
INDICATORS
SALMONELLA

	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	1000 mL	1000 mL		1000 mL	* mL	* mL	* grams					*

Preferred
Sampling
Container

See
text

See
text

Preservation
Technique

Pack
in ice

Pack
in ice

Operating
Range

Limit
Detection
Criteria

Std.
Dev.
for
in run
Duplicates

<20%
20-50%
50-100%

Precision

Units

* = Available upon request.

MICROBIOLOGICAL INDICATORS OF FECAL POLLUTION

ROUTINE

Total Coliforms (TC) Procedure

The TC group is comprised of all of the aerobic and facultative anaerobic, Gram-negative, non-sporeforming, rod-shaped bacteria which ferment lactose with gas formation within 48 hours at 35°C or all organisms which produce a colony with a gold-green metallic sheen within 24 hours of incubation using the MF technique. All coliforms are oxidase negative. This definition includes, in addition to the intestinal forms of the Escherichia coli group, closely related bacteria of the genera Klebsiella, Citrobacter and Enterobacter. Enterobacter - Citrobacter groups are common in soil but are also recovered from feces in small numbers. Therefore, the presence of total coliforms in water is not a specific indicator of sewage or fecal pollution but may also indicate soil runoff.

Sampling

Samples should be collected in pre-sterilized glass bottles with red labels (thiosulphate added) or in rubber syringes. Samples should be packed in ice but should not be frozen.

TC levels will undergo change even upon refrigeration; therefore, immediate delivery to the laboratory is essential. Analyses should be performed as soon as possible after collection, preferably within six hours, but not exceeding 24 hours (in some cases 3 days is allowed but this procedure is not advised). The necessary frequency of sampling is outlined in the MOE Water Management booklet (1978).

Method

Analysis for TC is by membrane filtration according to the MOE Handbook of Analytical Methods for Environmental Samples (HAMES). A given volume of the sample or dilution is filtered using a 0.45 µ gridded, white membrane filter (e.g. Gelman GN-6). The membrane is placed on m-Endo Agar LES (Difco) and incubated for 22 ± 2 hours at $35 \pm 0.5^\circ\text{C}$. After incubation, the number of total coliforms and background colonies are counted using a stereoscopic microscope at a 10X magnification.

Time of Analysis

The number of analyses performed per day will depend on the type of sample, the number of dilution steps required, the number of parameters requested per sample and the incubation periods for each parameter. TC results are generally available within 48 hours of submission.

FECAL POLLUTION INDICATORS	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg ^h	Bio Material	Dust Fall	Hi-Vol	Ambient Air
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TOTAL COLIFORMS

Minimum Sample Size	150 mL	150 mL		150 mL	* mL	* mL	* grams					*
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Preferred Sampling Container

See text

See text

Preservation Technique

Pack in ice

Pack in ice

Operating Range

Limit Detection Criteria

Std. Dev. for in run Duplicates

<20%
20-50%
50-100%

Precision

Units

* Available upon request

MICROBIOLOGICAL INDICATORS OF ORGANIC ENRICHMENT

Fungi (Yeasts and Molds)

Fungi are ubiquitously distributed, achlorophyllous, heterotrophic micro-organisms which are present wherever living or non-living organic matter occurs. Both saprophytic and pathogenic fungi have been found in sewage and sludge, domestic and industrial effluent and marine and freshwater environments. The densities and distribution of species of fungi in such habitats can be used as an index of the degree of organic enrichment. In addition, the presence of certain pathogenic fungi (e.g. *Canadida albicans*) in recreational waters is useful in assessing water quality and in evaluating public health hazards.

Sampling

Samples should be collected in pre-sterilized glass bottles with red labels (thiosulphate added) or in rubber syringes. Samples should be packed in ice but should not be frozen.

Fungal levels, yeasts in particular, will undergo change even upon refrigeration, therefore, immediate delivery to the laboratory is essential. Analyses should be performed as soon as possible after collection, preferably within six hours, but not exceeding 24 hours.

Method

No standard techniques have been developed for quantitative enumeration of fungi from an aquatic environment. In general, selective agar media, the membrane filtration technique and the spread plate technique are used. Fungal densities are expressed as colony-forming units (CFU) per 100 mL.

Time of Analysis

The number of analyses performed per day will depend on the type of sample, the number of dilution steps required and the incubation periods. In general, the analysis requires a five-day period. Prior consultation is required before this parameter can be requested.

ORGANIC ENRICHMENT INDICATORS FUNGI-YEASTS, MOLDS

	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	150 mL	150 mL		150 mL	*	*	* grams					*

Preferred Sampling Container

See text	See text		See text									
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Preservation Technique

Pack in ice	Pack in ice		Pack in ice									
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Operating Range

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Limit Detection Criteria

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Std. Dev. for in run Duplicates

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Precision

--	--	--	--	--	--	--	--	--	--	--	--	--

Units

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* = Available upon request

MICROBIOLOGICAL INDICATORS OF ORGANIC ENRICHMENT

Heterotrophic Bacteria - 20°C (HB-20) - Surface water

The HB-20 count is not a test for a specific microorganism. It is designed to enumerate as large a number as possible of those bacteria in water that require some organic carbon for their growth. The concentration of heterotrophic bacteria in water is affected by the levels of organic and inorganic nutrients present. Densities of HB-20 in lakes of high trophic status are higher than HB-20 levels in oligotrophic lakes.

Sampling

Samples should be collected in pre-sterilized glass bottles with red labels (thiosulphate added) or in rubber syringes. Samples should be packed in ice but should not be frozen.

HB-20 levels will undergo change even upon refrigeration; therefore, immediate delivery to the laboratory is essential. Analyses should be performed as soon as possible after collection, preferably within six hours, but not exceeding 24 hours.

Method

Analysis is by the spot plate or spread plate method. Two volumes of 0.1 mL of the sample and/or sample dilution are placed on pre-dried CPS agar plates. The spread plate method requires one volume of 0.1 to 1.0 mL spread over the surface of the pre-dried agar plate. Plates are incubated at $20 \pm 0.5^{\circ}\text{C}$ for 7 days. Using a stereoscope microscope (10X), a total colony count is obtained.

Time of Analysis

The number of analyses performed per day will depend on the type of sample, the number of dilution steps required, and the incubation period. HB-20 results by this method are available 8 days after sample submission.

ORGANIC ENRICHMENT INDICATORS HETEROTROPHIC BACTERIA - 20 °C (HB-20)

	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	150 mL	150 mL		150 mL								
Preferred Sampling Container	See text	See text		See text								
Preservation Technique	Pack in ice	Pack in ice		Pack in ice								
Operating Range												

Limit Detection Criteria

Std. Dev. for in run Duplicates
 <20%
 20-50%
 50-100%

Precision

Units

MICROBIOLOGICAL INDICATORS OF ORGANIC ENRICHMENT

Heterotrophic Bacteria - 35°C (HB-35)

The HB-35 parameter can be used for the analysis of treated waters (i.e. drinking waters, water in distribution systems, bottled water) and untreated well water. In these waters the HB-35 parameter is used to measure treatment efficiencies and as an indicator of bacterial water quality. Prior consultation is required before this test is requested.

Sampling

Samples should be collected in pre-sterilized glass bottles with red labels (thiosulphate added). Samples should be packed in ice but should not be frozen. HB-35 levels will undergo change even upon refrigeration; therefore immediate delivery to the laboratory is essential. Analyses should be performed as soon as possible after collection, preferably within six hours, but not more than 24 hours.

Method

Analysis is performed by filtering 1 mL through a membrane filter which is placed on standard plate count (mSPC) agar. Standard Methods (14th Edition) recommends an incubation period of 48 ± 3 hours (bottled water for 72 ± 4 hours) at $35 \pm 0.5^\circ\text{C}$. A total colony count which exceeds 500 organisms per mL is considered to represent poor water quality.

Time of Analysis

The number of analyses performed per day will depend on the type of sample, and the overall laboratory workload. HB-35 results by this method would be available 3 to 4 days after the sample is submitted.

ORGANIC
ENRICHMENT
INDICATORS
HETEROTROPHIC
BACTERIA - 35°C
(HB-35)

	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	150 mL	150 mL		150 mL	* mL	* mL						
Preferred Sampling Container	See text	See text		See text								
Preservation Technique	Pack in ice	Pack in ice		Pack in ice								

Operating
Range

Limit
Detection
Criteria

Std.
Dev.
for
in run
Duplicates

<20%
20-50%
50-100%

Precision

Units

* = Available upon request

**MICROBIOLOGICAL INDICATORS OF
SPECIFIC (e.g. INDUSTRIAL/AGRICULTURAL) POLLUTION**

***Klebsiella pneumoniae* (K. pneumoniae) - Non-routine Procedure**

K. pneumoniae are frequently found in the respiratory, intestinal and urogenital tracts of man, but these organisms may be isolated from a variety of animals and materials and are well distributed in the environment. It is associated primarily with water high in organic matter. Thus, K. pneumoniae can be used to determine the extent of influence of certain industrial wastes. A further concern, which still has to be proven epidemiologically, is that K. pneumoniae which is an opportunistic pathogen, could infect humans and cause a highly fatal type of pneumonia which accounts for 3% of bacterial pneumonias.

This is a newly developed technique which can only be performed on a limited number of samples. Prior consultation is required with the appropriate personnel in the Microbiology Section.

Sampling

Samples should be collected in pre-sterilized glass bottles with red labels (thiosulphate added) or in rubber syringes. Samples should be packed in ice but should not be frozen.

K. pneumoniae levels will undergo change even upon refrigeration; therefore, immediate delivery to the laboratory is essential. Analyses should be performed as soon as possible after collection, preferably within six hours, but not exceeding 24 hours.

Method

Analysis for Klebsiella pneumoniae is by membrane filtration of an appropriate volume or dilution of a sample with incubation on mK Agar at $35 \pm 0.5^{\circ}\text{C}$ for 48 ± 3 hours. Using a stereoscopic microscope (10X), all non-red colonies larger than 2 mm in diameter are counted as Klebsiella.

Time of Analysis

The number of analyses performed per day will depend on the type of sample, the number of dilution steps required, and the incubation period. K. pneumoniae results will be available 3 days after sample submission. Prior consultation is required before a request for analysis can be considered.

INDUSTRIAL-
AGRICULTURAL
POLLUTION
INDICATORS
K. PNEUMONIAE

Minimum Sample Size	150 mL	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'h	Bio Material	Dust Fall	Hi-Vol	Ambient Air
				150 mL	*	* mL	* grams					*

Preferred Sampling Container

See text

See text

Preservation Technique

Pack in ice

Operating Range

Limit Detection Criteria

Std. Dev. for in run Duplicates

<20%
20-50%
50-100%

Precision

Units

* = Available upon request.

MICROBIOLOGICAL INDICATORS OF SPECIFIC (E.G. INDUSTRIAL/AGRICULTURAL) POLLUTION

Nitrogen Cycle Bacteria

Several groups of bacteria contribute extensively to the transformations occurring within the nitrogen cycle, both in aquatic and terrestrial environments. These groups include: a) the ammonifiers, comprised mainly of heterotrophic bacteria, which release ammonia from decaying proteinaceous material; b) the nitrifying bacteria, comprised of the genera Nitrosomonas and Nitrobacter, which, by an autotrophic, aerobic process oxidize ammonia to nitrite and nitrite to nitrate; c) the denitrifying bacteria, a diverse bacterial group, which through mainly anaerobic processes, remove nitrate from the environment either by reduction to nitrite or ammonia, or by transformation of nitrate to nitrogen gas; and d) the nitrogen-fixing bacteria, i.e. Azotobacter, which transform nitrogen gas into ammonia nitrogen.

The detection and enumeration of these bacteria in water and wastewaters are not necessarily used as an indication of deteriorating water quality. However, elevated levels of nitrifying bacteria may indicate excessive loading with ammonia-nitrogen and an associated increased oxygen demand. Their enumeration, complemented by activity studies, reflects the potential of an aqueous environment to mineralize proteinaceous nitrogen (ammonifiers, nitrifiers and denitrifiers) or the ability to supplement nitrogen requirements through nitrogen fixation.

Methods presently available in the MOE are only for the enumeration of nitrifying and denitrifying bacteria.

Sampling

Samples should be collected in pre-sterilized glass bottles with red labels (thiosulphate added) or in rubber syringes. Samples should be packed in ice but should not be frozen.

Nitrogen cycle bacterial levels will undergo change even upon refrigeration; therefore, immediate delivery to the laboratory is essential. Analyses should be performed as soon as possible after collection, but not exceeding 48 hours.

Method

Nitrogen cycle bacteria are determined by MPN analyses in selective media broths. Incubation for the nitrifiers is at $28 \pm 0.5^{\circ}\text{C}$ for 28 days under aerobic conditions. Denitrifiers are incubated anaerobically at $28 \pm 0.5^{\circ}\text{C}$ from 7 to 21 days.

Time of Analysis

The number of analyses performed per day will depend on the type of sample, the number of dilution steps required, the number of parameters requested and the incubation periods for each parameter. Nitrosomonas and Nitrobacter results are available 29 days after sample submission. Denitrifier results are available in 8 to 22 days depending on the incubation time. Prior consultation with Microbiology Section staff is necessary before requesting these parameters.

INDUSTRIAL-
AGRICULTURAL
POLLUTION
INDICATORS
NITROGEN CYCLE
BACTERIA

	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Vegh	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	150 mL	150 mL		150 mL	*	*	*					*

Preferred
Sampling
Container

See
text

See
text

See
text

Preservation
Technique

Pack
in ice

Pack
in ice

Pack
in ice

Operating
Range

Limit
Detection
Criteria

Std.
Dev.
for
in run
Duplicates

<20%
20-50%
50-100%

Precision

Units

* = Available upon request.

MICROBIOLOGICAL INDICATORS OF SPECIFIC (e.g. INDUSTRIAL/AGRICULTURAL) POLLUTION

Phenol Degrading Bacteria

The phenol degrading bacteria are a small heterogeneous group of bacteria that can degrade phenols if the concentration does not exceed 2 g/L. These bacteria are not monitored in order to detect a microbial hazard but to help determine the possible consequences of pollution by phenol and related compounds.

Pollution of surface waters with phenolic substances occurs as a result of their extensive industrial use. The presence of phenolics in surface waters can pose a serious pollution threat due to the toxic nature of these compounds. The potential for serious damage to the environment is increased if the area affected does not contain significant concentrations of bacteria capable of degrading these chemicals.

Sampling

Samples should be collected in pre-sterilized glass bottles with red labels (thiosulphate added) or in rubber syringes. Samples should be packed in ice but should not be frozen.

The concentrations of phenol degrading bacteria will undergo change even upon refrigeration; therefore immediate delivery to the laboratory is essential. Analyses should be performed as soon as possible after collection, but not exceeding 24 hours.

Method

Phenol degrading bacteria levels are determined by MPN analyses in a broth containing phenol as the sole carbon source. Incubation is at $35 \pm 0.5^{\circ}\text{C}$ for 72 hours under aerobic conditions.

Time of Analysis

The number of analyses performed per day will depend on the type of sample, the number of dilution steps required, the number of parameters requested and the incubation periods for each parameter. Phenol degrading bacteria results are available four days after sample submission. Prior consultation before submitting samples is necessary.

INDUSTRIAL-
AGRICULTURAL
POLLUTION
INDICATORS
PHENOL DEGRADING
BACTERIA

Minimum Sample Size	150 mL	150 mL	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
			*	*	*					*
Preferred Sampling Container	See text	See text								
Preservation Technique	Pack in ice	Pack in ice								

Operating
Range

Limit
Detection
Criteria

Std.
Dev.
for
in run
Duplicates

<20%
20-50%
50-100%

Precision

Units

* = Available upon request.

**MICROBIOLOGICAL INDICATORS OF
SPECIFIC (e.g. INDUSTRIAL/AGRICULTURAL) POLLUTION**

Sulphate-Reducing Bacteria

The sulphate-reducing bacteria e.g. (*Desulfovibrio*, *Desulfotomaculum*) are a group of organisms which obtain energy from the reduction of sulphate to sulphide and grow in anaerobic environments which contain organics and a supply of sulphate. The activity of these organisms contributes to the deterioration of water quality by their production of H_2S and their capability of maintaining anaerobic and reducing environments.

The organisms have an economic importance in their contribution to the corrosion of iron pipes and steel containers, spoilage of stored petroleum products and the blackening of wood pulp.

Sampling

Samples should be collected in pre-sterilized glass bottles or rubber syringes. Bottles with thiosulphate added may be used but prior consultation with laboratory staff is requested.

Samples should be packed in ice but should not be frozen.

Sample containers should be as full as possible and tightly sealed to prevent oxidation. Do not leave an air space.

Sulphate reducer levels will undergo change even upon refrigeration; therefore, immediate delivery to the laboratory is essential. Analyses should be performed as soon as possible after collection, preferably within six hours, but not exceeding 48 hours.

Method

Sulphate reducers are determined by a Most Probable Number (MPN) technique. Using tubes of API broth with tryptone, a 3 x 3 series is set up using three appropriate volumes (weights) and/or dilutions of the sample (e.g. 3 tubes each of 10 mL, 1 mL and .1 mL). The tubes are layered with paraffin oil, to provide anaerobic conditions and incubated for 21 days at $28 \pm 0.5^\circ C$. A black precipitate is taken as a positive reaction.

Time of Analysis

The number of analyses performed per day will depend on the type of sample and the number of dilution steps required. Sulphate reducer results are available 22 days after sample submission. Prior consultation with Microbiology Section staff is required before this parameter can be requested.

INDUSTRIAL-
AGRICULTURAL
POLLUTION
INDICATORS
SULPHATE-REDUCING
BACTERIA

	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	150 mL	150 mL		150 mL	*	*	* grams					*
Preferred Sampling Container	See text	See text		See text								
Preservation Technique	Pack in ice	Pack in ice		Pack in ice								
Operating Range												

Limit
Detection
Criteria

Std.
Dev.
for
in run
Duplicates

Precision

Units

* = Available upon request.

**MICROBIOLOGICAL INDICATORS OF
SPECIFIC (e.g. INDUSTRIAL/AGRICULTURAL) POLLUTION**

Sulphur-Oxidizing Bacteria

The sulphur-oxidizing bacteria of the bacterial genus Thiobacillus are a group of aerobic, autotrophic organisms (T. thioparus, T. thiooxidans and T. ferrooxidans) which derive energy from the oxidation of elemental sulphur, sulphide or thiosulphate. These organisms are able to multiply and maintain acidic environments. They contribute to the deterioration of water quality by lowering the water pH when the water body is enriched with reduced sulphur compounds and are responsible for the formation of acidic conditions in leachates of mine-tailings containing sulphide-based ores. Their activity also contributes to the corrosion of concrete in sewers and structures immersed in water.

Sampling

Samples should be collected in pre-sterilized glass bottles or in rubber syringes. Bottles containing thiosulphate may be used but prior consultation with laboratory staff is required. Samples should be packed in ice but should not be frozen.

Sulphur oxidizer levels will undergo change even upon refrigeration; therefore, immediate delivery to the laboratory is essential. Analyses should be performed as soon as possible after collection, but not exceeding 48 hours.

Method

Sulphur-oxidizing bacteria are determined by a MPN method. Media most often used by the MOE are thioparus, thiooxidans and ferrooxidans broth. Incubation is under aerobic conditions at $28 \pm 0.5^{\circ}\text{C}$ for 21 days.

Time of Analysis

The number of analyses performed per day will depend on the type of sample and the number of dilution steps required. Sulphur oxidizer results are available 22 days after sample submission. Prior consultation with Microbiology staff is necessary before this parameter is requested.

INDUSTRIAL-
AGRICULTURAL
POLLUTION
INDICATORS
SULPHUR OXIDIZING
BACTERIA

	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	150 mL	150 mL		150 mL	* mL	* mL	* grams					*
Preferred Sampling Container	See text	See text		See text								
Preservation Technique	Pack in ice	Pack in ice		Pack in ice								
Operating Range												

Limit
Detection
Criteria

Std.
Dev.
for
in run
Duplicates

Precision

Units

* = Available upon request.

MICROBIOLOGICAL NUISANCE ORGANISMS

Certain microorganisms, although not pathogenic or indicative of the presence of fecal material, may be related to a deterioration in water quality or may reduce the efficiency of the effective treatment of water or sewage. Foul tastes and odours, slimes, discoloration, bulking of activated sludge, and the clogging or corrosion of treatment or distribution systems may all result from the presence of one or more groups of microorganisms. Nuisance organisms include iron bacteria such as Gallionella, Leptothrix, Sphaerotilus and Crenothrix, the sulphate reducing bacteria of the genera Desulfovibrio and Desulfotomaculum, the sulphur oxidizer Beggiatoa, actinomycetes, algae and fungi.

Sampling

Samples should be collected in pre-sterilized glass bottles with red labels (thiosulphate added). Samples should be packed in ice but should not be frozen. The sample should contain material representative of the problem such as slimes or filamentous growth collected aseptically.

Nuisance organism levels will undergo change even upon refrigeration; therefore, immediate delivery to the laboratory is essential. Analyses should be performed as soon as possible after collection, preferably within six hours, but not exceeding 48 hours.

Method

With the exception of the sulphate reducing bacteria and fungi, identification is generally based upon direct microscopic observation of the sample. Morphological characteristics are generally used to identify the organism(s) present. The sulphate reducers may be cultivated and their presence revealed by the detection of metabolic byproducts. Fungi may also be cultivated and later identified by microscopic observation of their morphological characteristics.

Time of Analysis

The number of analyses performed per day will depend on the type of sample, the number of parameters requested and the incubation periods (if necessary) for each parameter. Results of microscopic analyses are available two days after sample submission, however, because the method and number of analyses vary with the sample problem, some analyses may take one month to complete. Consultation with Microbiology staff is necessary prior to sample collection.

NUISANCE ORGANISMS	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
--------------------	---------------	----------------	---------	--------	-------------	--------	-----------------	-------	--------------	-----------	--------	-------------

Minimum Sample Size	150 mL	150 mL		150 mL	*	* mL	* grams					*
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Preferred Sampling Container	See text	See text		See text								
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Preservation Technique	Pack in ice	Pack in ice		Pack in ice								
------------------------	-------------	-------------	--	-------------	--	--	--	--	--	--	--	--

Operating Range												
-----------------	--	--	--	--	--	--	--	--	--	--	--	--

Limit Detection Criteria												
--------------------------	--	--	--	--	--	--	--	--	--	--	--	--

Std. Dev. for in run Duplicates												
---------------------------------	--	--	--	--	--	--	--	--	--	--	--	--

Precision												
-----------	--	--	--	--	--	--	--	--	--	--	--	--

Units												
-------	--	--	--	--	--	--	--	--	--	--	--	--

* = Available upon request

MOISTURE CONTENT

The moisture content may be determined on solid or semi-solid samples such as soils, sediments and sludges. On soil and sediment samples, the percent moisture is required for the conversion of results reported as percent dry weight to percent wet weight. For sludge samples, the test is performed as a routine process control at the treatment plant. The test results, while traditionally reported as percent moisture are in fact a measurement of all volatile material in the sample. The test detects the loss of that fraction of the water content which will evaporate in 4 hours at 105°C as well as any other constituents which volatilize under the same conditions. The result reported therefore is frequently higher than the actual moisture content of the sample.

Sampling

Sediment samples should be collected in wide-mouth glass bottles with moisture-tight PVC closures. Bottles should not be filled to more than 70% capacity to allow space for gas generation. Soil and vegetation samples may be collected in glass pomade jars with moisture tight caps. Samples should be tested within 2 or 3 days of collection and stored under refrigeration. This is very important for sludges because of their high bacterial activity.

Method

A weighed sample is heated to 105°C to drive off moisture. The weight loss on heating is reported as a percent fraction of the initial weight taken. Other volatile materials in the samples may be measured along with the moisture. There may be a weight loss in sludges due to decomposing organic materials.

Time of Analysis

Approximately 30 samples may be analyzed per 24 hour period.

MOISTURE CONTENT	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Vegh	Bio Material	Dust Fall	Hi-Vol	Ambient Air
---------------------	------------------	-------------------	---------	--------	----------------	--------	--------------------	------	-----------------	--------------	--------	----------------

Minimum
Sample
Size

10
grams

Preferred
Sampling
Container

Glass
bottle
see
text

Preservation
Technique

4°C

Operating
Range

0
to
100

Limit
Detection
Criteria

Std.
Dev.
for
in run
Duplicates

<20%
20-50%
50-100%

Precision

Units

Percent

Percent

Percent

MOLYBDENUM

Molybdenum is found in nature as the minerals molybdenite, wulfenite, and molybdenum ochre. Its industrial applications include the manufacture of steel alloys, glass, ceramics, pigments, lacquers, paints, electrical wiring, and fertilizer. It is also used as a catalyst in certain chemical processes. Its presence in natural waters is likely the result of industrial discharges and agricultural run off as well as weathering processes.

In plants, molybdenum is believed to have an important influence on the ascorbic acid (vitamin C) equilibrium. It is essential to the nutrition of leguminous crops and all other nitrogen-fixing organisms. In animals, the function of molybdenum is not well understood but it appears to behave much like copper. It is known to be a constituent of certain flavoprotein enzymes which have essential metabolic functions.

Of all the industrial heavy metals, molybdenum is one of the least toxic. Isolated instances of molybdenum poisoning are generally associated with cattle grazing in pastures where the soil contains elevated levels of molybdenum (20 - 100 mg/l). Normal soils contain between 3 - 5 mg/l. This condition in cattle is commonly known as "teart" and is characterized by severe diarrhea, anemia, and harsh discolored fur.

Molybdenum is rarely encountered in domestic water supplies and no limit has been specified for its maximum acceptable concentration. Levels should not exceed 0.010 mg/L for irrigation waters used continuously on all soil.

Sampling

Special acid washed plastic containers are required for trace level determinations in domestic water and surface water samples. Glass containers are required for domestic waste and industrial waste samples. Add 20 drops of HNO_3 per liter. Pomade jars should be used for soil and sediment samples. Vegetation samples should be collected in plastic bags and refrigerated. Fish samples should be placed in plastic bags and frozen.

Method

Samples are digested with an appropriate acid mixture and analyzed by atomic absorption. Sewage samples are analyzed by ICP atomic emission.

Time of Analysis

Approximately 50 molybdenum analyses can be performed per day.

MOLYBDENUM	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	400 mL	400 mL	25 mL	50 mL	100 mL	10 mL	2 grams	2 grams	50 grams	200 mL		
Preferred Sampling Container	Plastic bottle Acid washed	Plastic bottle Acid washed	Plastic bottle Acid washed	Glass bottle	Glass bottle	Glass bottle	Pomade jar	Plastic bag	Plastic bag	Sampling jar		
Preservation Technique	HNO ₃ pH <2	HNO ₃ pH <2	HNO ₃ pH <2	HNO ₃ pH <2	HNO ₃ pH <2	None	None	4 °C	Freeze	None		
Operating Range	0.002 to 0.25	0.002 to 0.25	* 0.002 to 0.25	0.05 to 70	0.02 to 2.5	0.25 to 350	2 to 2000	2 to 10	*	0.02 to 3.0		
Limit Detection Criteria	0.002	0.002		0.05	0.02	0.25	2	2		0.02		
Std. Dev. for in run Duplicates												
Precision	0.02 at 0.20	0.02 at 0.20		**	0.03 at 0.20	**	0.27 at 2.2	-		-		
Units	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	μg/g	μg/g	μg/g	μg/m ² /30 days		

* = Test available upon request
 ** = ± 10% at 10x Detection limit.

MUSKS

Musks are used to test for and track leaks in storage tanks for gasoline and other petrohydrocarbons. A musk solution is poured into a storage tank and then several days later holes are dug around the tank and tested for the presence of the musk.

Musk solutions used are musk ambrette, musk xylol, musk ketone, musk tibetene and moskene.

Sampling

Aqueous samples should be collected in 1 liter glass bottles with foil lined caps. Samples should be kept cool and transported to the laboratory as quickly as possible.

Method

Samples are analyzed by gas chromatography after solvent extraction of the musks.

MUSKS	Gasoline	Fuel Oil
Minimum Sample Size	1000 mL	1000 mL
Preferred Sampling Container	Glass bottle	Glass bottle
Preservation Technique	4°C	4°C
Operating Range	>50	>50
Limit Detection Criteria	50	50
Std. Dev. for in run Duplicates	<20% 20-50% 50-100%	
Precision	*	*
Units	ng/L	ng/L

* = Replicate analyses should be within 10% of the mean.

NICKEL

Nickel is more abundant in Ontario than anywhere else in the world. It is found primarily as the sulphide minerals pentlandite and pyrrhotite, as well as the arsenic mineral kupfernickel. Nickel has numerous industrial applications; the most important of which is the manufacture of high quality heat and corrosion resistant steel. Electroplating processes which utilize nickel are also widespread. Levels in natural waters are very low; therefore, if high nickel levels are found, they are likely the result of an industrial discharge or mine drainage. The median freshwater concentration of nickel in North American Rivers is 0.10 mg/L.

Nickel and its salts have generally proven to be non-toxic to man even at very high levels. Nickel sulphate and nickel bromide have even been used therapeutically for headache, neuralgia, and insomnia. However, nickel carbonyl is suspected of being carcinogenic although confirmatory evidence is not well established. Contact with the metal can cause a severe form of dermatitis. The element appears to be biologically active, activating several enzyme systems, and appearing in high concentrations in ribonucleic acid. Toxicity to plants and invertebrates varies with the species. Acute nickel toxicity to fish is generally less than that of copper or zinc. The 48 hour LC_{50} for rainbow trout is 32 mg/L while it is 0.750 mg/L for copper and 4 mg/L for zinc. Concentrations of nickel in an unfiltered sample should not exceed 0.025 mg/L to protect aquatic life. Irrigation waters which are used continuously on all soil should not contain more than 0.20 mg/L.

Sampling

Special acid washed plastic containers are required for trace level determinations in domestic water and surface water samples. Glass containers are required for domestic waste and industrial waste samples. Add 20 drops of HNO_3 per liter. Pomade jars should be used for soil and sediment samples. Vegetation samples should be collected in plastic bags and refrigerated.

Method

Samples are digested in the appropriate acid mixture and analyzed. Sewage, sludge and Hi-Vol samples are analyzed by ICP atomic emission. All other samples are analyzed by atomic absorption.

Time of Analysis

Approximately 50 nickel analyses can be performed per day.

NICKEL	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	400 mL	400 mL	25 mL	50 mL	100 mL	10 mL	2 grams	2 grams	50 grams	200 mL	1 filter strip	
Preferred Sampling Container	Plastic bottle Acid washed	Plastic bottle Acid washed	Plastic bottle Acid washed	Glass bottle	Glass bottle	Glass bottle	Pomade jar	Plastic bag	Plastic bag	Sampling jar	Envelope	
Preservation Technique	HNO ₃ pH <2	HNO ₃ pH <2	HNO ₃ pH <2	HNO ₃ pH <2	HNO ₃ pH <2	None	None	4°C	Freeze	None	None	
Operating Range	0.002 to 0.25	0.002 to 0.25	0.001 to 0.200	0.05 to 70	0.02 to 2.5	0.25 to 350	3 to 1000	1 to 100	0.25 to 25.0	0.06 to 2.0	0.0002 to 0.35	
Limit Detection Criteria	0.002	0.002	0.001	0.05	0.02	0.25	3	1	0.25	0.06	0.0002	
Std. Dev. for in run Duplicates												
Precision	0.002 at 0.025	0.002 at 0.025	0.0006 at 0.006	*	0.02 at 0.25	*	1.71 at 52.5	2.5 at 50	-	-	*	
Units	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	μg/g	μg/g	μg/g	μg/m ² /30 days	μg/m ³	

* = ± 10% at 10x Detection Limit.

NITROGEN - AMMONIA AND AMMONIUM

Ammonia nitrogen is commonly encountered in domestic wastewater and industrial effluents. It is also found as a biochemical reduction product in ground water. Other sources include fertilisers, cleaning compounds and atmospheric precipitation.

Ammonia nitrogen may be toxic to fish because it reduces the oxygen carrying capacity of the blood. Ammonia toxicity is related to the amount of ammonium ion and is dependent on pH, temperature and dissolved oxygen. Ammonia also exerts a high oxygen demand when converted to nitrite and nitrate, and interferes with water treatment procedures by reacting with chlorine to form chloramines. The latter reaction greatly reduces the effectiveness of the chlorination process. It is also objectionable because its nutritive properties promote the excessive growth of algae and other aquatic plants. Ammonia is rarely found in concentrations high enough to be harmful to humans. Concentrations in surface waters should not exceed 0.02 mg/L of unionized ammonia for the protection of aquatic organisms.

Ammonia results are of great value in interpreting the effectiveness of the waste stabilization process at sewage treatment plants. The relative abundance of ammonia in the treated water is an indication of how far treatment has progressed.

Ammonia is rather short lived in surface waters; therefore, it may not reveal the complete extent of pollution. Results must be interpreted with full allowance for its perishability, both in situ and in the sample following collection.

Sampling

Ammonia is unstable and rapidly converted to other nitrogen compounds by bacterial activity. Samples must be refrigerated or frozen immediately after collection and delivered to the laboratory as soon as possible. Aqueous samples should be collected in glass or polystyrene bottles.

Method

Samples are pretreated by filtration and analysis is accomplished colorimetrically by the alkaline phenol-hypochlorite method using an AutoAnalyzer system. Sludge analysis involves use of a distillation unit in addition to the AutoAnalyzer system. Water or KCl extracts are used for soils and sediments. High levels of manganese, cobalt, iron, trivalent chromium, and calcium are known to produce interferences. Ammonia results are reported in mg/L of nitrogen.

Time of Analysis

Approximately 200 ammonia analyses on aqueous samples may be performed during an 18 hour AutoAnalyzer working period.

NITROGEN-AMMONIA AND AMMONIUM	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'h	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	75 mL	75 mL	75 mL	75 mL	75 mL	75 mL	2 grams				1 filter strip	
Preferred Sampling Container	Glass or polystyrene	Glass or polystyrene	Polystyrene	Glass bottle	Glass bottle	Glass bottle	Pomade jar				Envelope	
Preservation Technique	Freeze or 4°C	Freeze or 4°C	Freeze or 4°C	4°C	4°C	4°C	Freeze or 4°C				None	
Operating Range	0.007 to 0.400	0.2 to 20	0.007 to 0.400	0.2 to 20	0.2 to 20	<1000	3 to 200				-	
Limit Detection Criteria	0.014	0.382	0.014	0.382	0.382	-	5.8				-	
Std. Dev. for in run Duplicates	0.0043 0.0065 0.0043	0.116 0.177 0.219	0.0043 0.0065 0.0065	0.116 0.177 0.219	0.116 0.177 0.219	- 12.2 12.8	-					
Precision												
Units	mg/L Nitrogen	mg/L Nitrogen	mg/L Nitrogen	mg/L Nitrogen	mg/L Nitrogen	mg/L Nitrogen	µg/g				µg/m ³ as Nitrogen	

NITROGEN - NITRATE

Nitrate is formed via the oxidation of nitrite by autotrophic nitrifying bacteria and represents the most highly oxidized form of nitrogen in the nitrogen cycle. It is generally found at trace levels in all surface waters but may become very high in ground water as a result of soil leaching. Potential sources of nitrates in water arise from the use of fertilisers, nitrogen fixation of micro-organisms and plants, decomposition of sewage wastes, leaching from soils and rocks and acidic precipitation. Nitrate provides an alternate source of oxygen when the dissolved oxygen has been depleted.

Nitrate results are of value in interpreting the effectiveness of the waste stabilization process at sewage treatment plants. Its abundance in treated water is an indication of how far treatment has progressed. Biochemical reduction of nitrate may frequently result in the production of nitrogen gas rather than ammonia (denitrification). This reaction is undesirable during the activated sludge process since the nitrogen gas tends to buoy the sludge and thereby inhibit digestion.

Nitrates are objectionable because their nutritive properties promote the excessive growth of algae and other aquatic plants. Although nitrates are considered non-toxic to adults, high levels in domestic water supplies contribute to a condition known as infant methemoglobinemia in which the oxygen carrying capacity of the blood is inhibited. The maximum acceptable level for domestic water supplies in Ontario is 10 mg/L of nitrate as nitrogen if the water is to be used for infant feeding. Livestock water should not contain more than 100 mg/L of nitrate plus nitrite, reported as nitrogen.

Sampling

Nitrate is unstable and is rapidly converted to other nitrogen compounds by bacterial action. Refrigeration is helpful but freezing is the preferred form of preservation. Aqueous samples should be collected in glass or polystyrene bottles. Soil samples should be collected in pomade jars.

Method

Precipitation samples are analyzed by ion chromatography.

Other aqueous samples are filtered and the nitrate is reduced to nitrite with hydrazine sulphate. The nitrite then undergoes a diazotisation reaction with sulphanilamide and N(1-naphthyl) ethylenediamine dihydrochloride. The analysis is performed on an AutoAnalyzer system. Solid samples undergo extraction with water or potassium chloride before colorimetric analysis. High levels of copper, iron calcium, magnesium and various oxidizing agents are known to interfere with the analysis.

Time of Analysis

Approximately 200 nitrate analyses can be performed per day colorimetrically on aqueous samples.

NITROGEN NITRATE	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	75 mL	75 mL	75 mL	75 mL	75 mL	75 mL	2 grams	grams	grams	200 mL	1 filter strip	
Preferred Sampling Container	Glass or polystyrene	Glass or poly- styrene	Poly- styrene	Glass bottle	Glass bottle	Glass bottle	Pomade jar			Sampling jar	Manila envelope	
Preservation Technique	Freeze or 4°C	Freeze or 4°C	Freeze or 4°C	4°C	4°C	4°C	Freeze or 4°C			None	None	
Operating Range	0.01 to 1.00	0.1 to 20.0	0.03 to 1.00	0.1 to 20.0	0.1 to 20.0	0.1 to 20.0	1 to 200			250 to 50,000	0.05 to 9.0	
Limit Detection Criteria	0.020	0.217	0.066	0.217	0.217	0.217	2.17			250	0.05	
Std. Dev. for in run Duplicates	0.0060 0.0071 0.0089	0.066 0.120 0.272	0.020 0.012 0.016	0.066 0.120 0.272	0.066 0.120 0.272	0.066 0.120 0.272						
Precision	-	-	-	-	-	-	-			-	0.2 at 5	
Units	mg/L Nitrogen	mg/L Nitrogen	mg/L Nitrogen	mg/L Nitrogen	mg/L Nitrogen	mg/L Nitrogen	µg/g Nitrogen			µg/m ² / 30 days	µg/m ³ Nitrogen	

NITROGEN - NITRITE

Nitrite is present in natural waters as an intermediary in the oxidation of ammonia by autotrophic nitrifying bacteria or in the anaerobic reduction of nitrate. In either case, its presence is short lived with the concentration seldom exceeding 1.0 mg/L as nitrogen. Higher concentrations are indicative of an industrial effluent. Waters containing nitrite are in the midst of rapid chemical changes.

Nitrite results may be used in interpreting the effectiveness of the waste stabilization processes at sewage treatment plants, but these results must be interpreted with full allowance for extreme perishability. The usefulness of nitrite results is questionable since it is highly unlikely that the concentration at the time of analysis represents the concentration at the time of sampling.

Nitrite is more toxic than nitrate. Livestock water should not contain more than 10.0 mg/L as nitrogen.

Sampling

Nitrite is unstable and is rapidly converted to other nitrogen compounds by bacterial action. Refrigeration is helpful but freezing is the preferred form of preservation. Immediate delivery to the laboratory is essential. Aqueous samples should be collected in glass or polystyrene bottles. Soil samples should be collected in pomade jars.

Method

Aqueous samples are pretreated by filtration and analysis is accomplished colorimetrically by a diazotisation reaction of nitrite with sulphanilamide and N(1-naphthyl) ethylene diamine dihydrochloride. The colorimetry portion of the procedure is performed with an AutoAnalyzer system. Solid samples undergo extraction with water or potassium chloride before colorimetric analysis. High levels of free chlorine, nitrogen trichloride and colored substances may interfere. Results are reported as mg/L of N.

Time of Analysis

Approximately 200 nitrite analyses can be performed per day.

NITROGEN NITRITE	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	75 mL	75 mL		75 mL	75 mL		2 grams					
Preferred Sampling Container	Glass or poly- styrene	Glass or poly styrene		Glass bottle	Glass bottle		Pomade jar					
Preservation Technique	Freeze or 4°C	Freeze or 4°C		4°C	4°C		Freeze or 4°C					
Operating Range	0.001 to 0.100	0.02 to 2.00		0.02 to 2.00	0.02 to 2.00		0.2 to 20					
Limit Detection Criteria	0.002	0.030		0.030	0.030		0.3					
	0.001	0.015		0.015	0.015		0.15					
Std. Dev. for in run Duplicates	<20% 0.0006 0.0008 0.0017	0.009 0.0204 0.0244		0.009 0.0204 0.0244	0.009 0.0204 0.0244		-					
Precision												
Units	mg/L Nitrogen	mg/L Nitrogen		mg/L Nitrogen	mg/L Nitrogen		µg/g Nitrogen					

NITROGEN - TOTAL KJELDAHL

Total Kjeldahl nitrogen is a measure of all the nitrogen in the trinegative state. It is primarily a measure of nitrogen present in the organic form (soluble or particulate) such as proteins, amines and urea as well as nitrogen present as free ammonia. It does **not** include nitrogen present as nitrite or nitrate. The organic nitrogen fraction can be estimated by taking the difference between the free ammonia and the total Kjeldahl nitrogen results.

Kjeldahl nitrogen results are of great value in interpreting the effectiveness of the waste stabilization process at sewage treatment plants. Treatment follows a natural sequence of biochemical reactions in which nitrogen components are successively oxidized from organic nitrogen to ammonia, nitrite and then nitrate and nitrogen gas. The relative abundance of each of the organic forms in the effluent is an indication of how far the treatment has progressed.

Sewage and industrial waste treatment effluents and runoff from agricultural areas, where fertilisers have been used, are the primary sources of TKN besides the natural nitrogen fixation and anaerobic nitrate reduction processes. There are no specific guidelines for total Kjeldahl nitrogen in drinking water although organic nitrogen levels should not exceed 0.15 mg/L.

Sampling

Kjeldahl nitrogen is slowly converted to other nitrogen compounds by bacterial activity in the presence of moisture. For aqueous samples, if representative results are expected, analyses should be performed within two or three days after sampling. Aqueous samples must be refrigerated or frozen immediately after collection and delivered to the laboratory as soon as possible. Aqueous samples should be collected in glass or polystyrene bottles.

Soil and vegetation samples should be collected in pomade jars and plastic bags respectively and both should be refrigerated and dried as soon as possible.

Method

Aqueous samples are pre-treated by digestion with sulphuric acid, mercuric oxide and potassium sulphate. Analysis is accomplished colorimetrically by the alkaline phenol hypochlorite method adapted to an Auto-Analyzer system. High levels of manganese, cobalt, iron, trivalent chromium and calcium are known to cause interferences. Aqueous Kjeldahl nitrogen results are reported in mg/L of nitrogen. Solid samples are pretreated by digestion with sulphuric acid, mercuric oxide and potassium persulphate.

Results for solid samples are reported as mg/g as nitrogen.

Time of Analysis

Approximately 200 samples may be analyzed during an 18 hour Auto-Analyzer working period.

NITROGEN TOTAL KJELDAHL	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'h	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	75 mL	75 mL		75 mL	75 mL	75 mL	1.0 grams	0.5 grams				
Preferred Sampling Container	Glass or poly- styrene	Glass or poly- styrene		Glass bottle	Glass bottle	Glass bottle	Pomade jar	Plastic bag				
Preservation Technique	Freeze or 4°C	Freeze or 4°C		4°C	4°C	4°C	4°C and dry	4°C and dry				
Operating Range	0.04 to 2.00	0.7 to 10.0		0.7 to 10.0	0.7 to 10.0	0.7 to 10.0	0.04 to 6.4	0.25 to 40				
Limit Detection Criteria	0.088	1.35		1.35	1.35	1.35	0.04	0.25				
Std. Dev. for in run Duplicates	0.027 0.036 0.039	0.411 0.117 0.090		0.411 0.117 0.090	0.411 0.117 0.090	0.411 0.117 0.090						
Precision							0.21 at 3	1.4 at 20				
Units	mg/L Nitrogen	mg/L Nitrogen		mg/L Nitrogen	mg/L Nitrogen	mg/L Nitrogen	mg/g Nitrogen	mg/g Nitrogen				

NITROTOLUENES

Nitrotoluenes are analyzed to check the purity of fluoridation agents at water treatment plants. Compounds analyzed are ortho-, meta-, para-nitrotoluene, 2,4 di-nitrotoluene and 3,4-dinitrotoluene. These compounds are used mainly in organic synthetic reactions and in the manufacture of dyes.

Inhalation of vapours may cause difficulty in breathing, cyanosis and even unconsciousness. If taken internally, they may damage the eyes and cause the same effects as by inhalation.

Nitro aromatics tend to be very potent mutagenic and carcinogenic compounds in the Ames test.

Sampling

Aqueous samples should be collected in 1 liter glass bottles with foil lined caps. Samples should be kept cool and transported to the laboratory as quickly as possible.

Method

Samples are analyzed by gas chromatography following extraction with an appropriate solvent.

NITROTOLUENES	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	1000 mL	1000 mL		1000 mL	1000 mL							
Preferred Sampling Container	Glass bottle	Glass bottle		Glass bottle	Glass bottle							
Preservation Technique	4° C	4° C		4° C	4° C							
Operating Range	>0.5	>0.5		>0.5	>0.5							
Limit Detection Criteria	0.5	0.5		0.5	0.5							
Std. Dev. for in run Duplicates												
Precision	*	*		*	*							
Units	µg/L	µg/L		µg/L	µg/L							

* = Replicate analyses should be within 10% of the mean.

ORGANIC SOLVENTS

Scans can be performed on samples to detect the presence of organic solvents. By these scans the presence of chlorinated hydrocarbons, aromatic hydrocarbons, ketones, alcohols, etc. can be detected.

Sampling

Aqueous samples should be collected in 1 liter glass bottles with foil lined tops. Solid samples should be collected in pomade jars. Air samples should be collected in polyester protected, aluminized bags. Aqueous and solid samples should be kept cool and transferred to the laboratory as quickly as possible. Bags should be stored at room temperature and in darkness.

Method

Samplers are directed to the individual outlines for descriptions of the analyses.

ORGANIC SOLVENTS	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	1000 mL	1000 mL		1000 mL	1000 mL	20 grams	20 grams	20 grams	20 grams			Half full bag
Preferred Sampling Container	Glass bottle	Glass bottle		Glass bottle	Glass bottle	Glass bottle	Pomade jar	Pomade jar	Pomade jar			Sampling bag
Preservation Technique	4°C	4°C		4°C	4°C	4°C	4°C	4°C	4°C			Keep in dark place
Operating Range												

SEE INDIVIDUAL OUTLINES

FOR DETAILS

Std. Dev. for in run Duplicates
< 20%
20-50%
50-100%

Precision

Units

ORGANOCHLORINE PESTICIDES AND POLYCHLORINATED BIPHENYLS

The organochlorine pesticides (OC's) form a class of compounds which includes widely used insecticides such as DDT. Polychlorinated biphenyls (PCB's) are not used as pesticides but have many industrial uses.

OC's can be classified as halogen derivatives of alicyclic hydrocarbons (BHC, lindane, heptachlor, aldrin, dieldrin, endrin) or aromatic hydrocarbons (DDT, DDD, DDE, endosulfan, HCB, methoxychlor).

The possibility of environmental contamination by organochlorine pesticides and polychlorinated biphenyls is of particular concern in view of their persistence. The stability of these products allows them to persist in the environment indefinitely creating a special hazard to wildlife due to the capability of many organisms to concentrate these compounds. This means that low levels (ng/L) of OC's and PCB's in water can be biomagnified up the food chain so that fish and fish-eating birds may contain mg/kg levels of these products. PCB's and DDT have been implicated in egg shell thinning in wild birds, endangering survival of the species.

Generally, OC's are resistant to degradation and oxidation, are relatively insoluble in water, have low volatility, and are lipophilic. Similarly, PCB's are inert, have low volatility and are insoluble in water, however, they are also resistant to acids, alkalis and high temperatures. PCB's are used in protective coatings, plasticizers, water proofing compounds, asphalts, inks, casting waxes, adhesives and as dielectrics, hydraulic fluids, grinding fluids and high pressure lubricants. Generally, the acute toxicity of organochlorine pesticides is low except for aldrin, endrin, toxaphene and endosulfan where the LD_{50} is in the 12 - 50 mg/kg range. Maximum concentrations for the protection of aquatic life in unfiltered samples are: 0.001 $\mu\text{g/L}$ for Aldrin, Dieldrin and Heptachlor; 0.003 $\mu\text{g/L}$ for DDT and metabolites; 0.002 $\mu\text{g/L}$ for Endrin and 0.01 $\mu\text{g/L}$ for Lindane. The edible portion of fish should not exceed 0.3 $\mu\text{g/g}$ of aldrin, dieldrin, endrin, heptachlor or lindane for the protection of human consumers of fish. Drinking water levels are 17.0 $\mu\text{g/L}$ for Aldrin and Dieldrin, 18.0 $\mu\text{g/L}$ for Heptachlor, 1 $\mu\text{g/L}$ for Endrin, 42 $\mu\text{g/L}$ for DDT's and 56 $\mu\text{g/L}$ for Lindane.

PCB's should not exceed 0.001 $\mu\text{g/L}$ in an unfiltered sample to protect aquatic life and 3 $\mu\text{g/L}$ in drinking water.

Sampling

Water, oil, soil, and vegetation samples should be collected in solvent rinsed and dried brown glass bottles or jars with foil lined caps. Plastic bottles should not be used as plasticizers may leach into the sample and interfere with the analysis. All these samples should be refrigerated immediately at 2 - 5°C. Fish samples should be wrapped in solvent rinsed aluminum foil and frozen.

Method

A sample is solvent extracted, dried, concentrated and cleaned up to remove interferences. The clean extract is examined by electron capture gas chromatography. For water samples, if no PCB's are present, pesticides are determined directly on the clean extract fractions by standard calibration and peak height quantitation. PCB's in fish, soil, sediment and water samples are separated from organochlorine pesticides by Florisil column chromatography, enabling the determination of each separately as above.

Time of Analysis

Approximately 15-20 analyses may be performed per day.

ORGANO-CHLORINE PESTICIDES and PCB'S											
	Surface Water	Drinking Water	Precip. Snow	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material (Fish)	Oil	Ambient Air
Minimum Sample Size	800 mL	800 mL	800 mL	800 mL	800 mL	800 mL	10 grams	10 grams	10 grams	10 mL	10 m ³
Preferred Sampling Container	Special glass bottle	Special glass bottle	Special glass bottle	Special glass bottle	Special glass bottle	Special glass bottle	Pomade jar see text	Pomade jar see text	Aluminum foil	Pomade jar	Florisil absorbant tube
Preservation Technique	4 ^o C	4 ^o C	4 ^o C	4 ^o C	4 ^o C	4 ^o C	4 ^o C	4 ^o C	Freeze	4 ^o C	4 ^o C
Operating ** Range	>0.001	>0.001	>0.001	>0.001	>0.001	>0.001	>0.001	>0.001	>0.001	>5	>0.01
Limit Detection Criteria	0.001 to 0.02	0.001 to 0.02	0.001 to 0.02	0.001 to 0.02	0.001 to 0.02	0.001 to 0.02	0.001 to 0.02	0.001 to 0.02	0.001 to 0.02	5 to 100%	0.01
Std. Dev. for in run Duplicates											
Precision	*	*	*	*	*	*	*	*	*		*
Units	µg/L	µg/L	µg/L	µg/L	µg/L	µg/g	µg/g	µg/g	µg/g	µg/g	µg/m ³

* = Replicate analyses must be within 10% of the mean.

** = Actual operating range may vary from the detection limits shown to percent levels.

ORGANO HALIDES - VOLATILE

Chloroform is produced mainly by methane chlorination. Chloroform is used to produce chlorodifluoromethane, a refrigerant and coolant in air conditioning. Chloroform is also used as a mildew preventative for tobacco seedlings, for soil fumigation and as an extractant for nicotine, penicillin, essential oils and alkaloids. The powerful anesthetic action of chloroform is associated with cumulative toxic effects, primarily on the liver. Inhalation of concentrations of 0.03 - 0.04% can be fatal in 30 minutes. A maximum safe concentration is 0.001%. Carbontetrachloride is manufactured mainly by the chlorination of methane. The principal uses are in fluorinated refrigerant and propellant manufacture, fire extinguishers, and formerly in dry cleaning. Inhalation of CCl_4 vapour can produce unconsciousness in 1 - 2 minutes. Repeated exposure to levels about 100 ppm can produce chronic toxic effects. Carbontetrachloride and chloroform are both carcinogenic to mice through oral administration.

Methylene chloride is used mainly in solvent applications such as paint strippers, but also is used as a propellant and a refrigerant. Methylene chloride forms toxic compounds such as phosgene when exposed to hot surfaces or open flames. It is the least toxic of the chloromethanes.

Tri- and tetra-chloroethylene are important solvents used in dry cleaning and fumigation. While these compounds are widely used, toxicity data is limited.

Other compounds which can be detected include vinyl and methyl chloride, bromodichloromethane and chlorodibromomethane.

Sampling

Aqueous samples should be collected in 1 liter glass bottles with foil lined caps. Samples should be refrigerated and should be transported to the laboratory as quickly as possible.

Method

Aqueous samples are generally analyzed by gas chromatography using direct injection. Methyl chloride must be extracted with a solvent and analyzed using head space analysis.

ORGANO- HALIDES VOLATILE	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
	1000 mL	1000 mL	1000 mL	1000 mL	1000 mL	grams	grams	grams	grams			
Minimum Sample Size	Glass bottle	Glass bottle	Glass bottle	Glass bottle	Glass bottle							
Preferred Sampling Container	4°C	4°C	4°C	4°C	4°C							
Preservation Technique	>1.0	>1.0	>1.0	>1.0	>1.0							
Operating Range	1.0	1.0	1.0	1.0	1.0							
Limit Detection Criteria												
Std. Dev. for in run Duplicates												
Precision	*	*	*	*	*							
Units	µg/L	µg/L	µg/L	µg/L	µg/L							

* = Replicate analyses should be within 10% of the mean.

ORGANOPHOSPHORUS INSECTICIDES

Organophosphorus insecticides have gained widespread useage in orchards, nurseries and greenhouses. Because of their low persistency and high effectiveness, they are now used world wide. About 140 phosphorus compounds are or were used as practical pesticides or plant growth regulators.

This test now detects dichlorvos, phorate, diazinon, parthion, methylparathion, malation, ethion, methyltrithion, dursban and reldan.

Due to their ester nature, many of these compounds may be metabolized into inorganic phosphates in living organisms. Toxicity in humans is a result of deactivating cholinesterase in the body. This allows uninterrupted pulses to flow along the nervous system, a condition which will eventually result in death. Levels should never exceed 100 $\mu\text{g/L}$ in raw water supplies or in livestock water supplies. Levels which can be harmful to aquatic life vary from 0.001 $\mu\text{g/L}$ for dichlorvos to 0.08 $\mu\text{g/L}$ for diazinon. The Environmental Protection Service has given the following ratings for Ecological Hazards: Extreme - phorate and dursban; High - diazinon, malathion and ethion; Moderate - dichlorvos and parathion.

Sampling

Aqueous samples should be collected in solvent rinsed, brown glass bottles with foil lined caps. Bottles should be filled to the mark. Solid samples should be collected in solvent rinsed jars. All samples should be refrigerated and transferred to the laboratory as quickly as possible.

Method

Samples undergo solvent extraction, Florisil clean-up and analysis by element selective detector gas chromatography.

Time of Analysis

Approximately 6 analyses can be performed per day.

ORGANO PHOSPHORUS INSECTICIDES

Surface Water Drinking Water Precip. Sewage Ind. Wastes Sludge Soil & Sediment Vegh Bio Material Dust Fall Hi-Vol Ambient Air

Minimum Sample Size 800 mL 800 mL 800 mL 800 mL 800 mL 10 grams 10 grams Special jar

Preferred Sampling Container Special bottle Special bottle Special bottle Special bottle Special jar Special jar

Preservation Technique 4°C 4°C 4°C 4°C 4°C 4°C 4°C

Operating ** Range >0.01 >0.01 >0.01 >0.01 >0.01 >0.001 >0.001

Limit Detection Criteria 0.01 to 0.10 0.01 to 0.10 0.01 to 0.10 0.01 to 0.10 0.001 to 0.01 0.001 to 0.01

Std. Dev. for in run Duplicates <20% 20-50% 50-100%

Precision * * *

Units µg/L µg/L µg/L µg/g µg/g

* = Replicate analyses should be within 10% of the mean.

** = Actual operating range may vary from the detection limits shown to percent levels.

ORGANO SULPHUR COMPOUNDS

The organo sulphur compounds analyzed in this group are carbon disulphide, carbonyl sulphide, dimethylsulphide, dimethyldisulphide, diethyl sulphide, thiophene and mercaptans such as methyl-, ethyl-, n-propyl-, isopropyl-, n-butyl- and isobutyl-mercaptan. Most of these compounds have a very disagreeable odour associated with them. The major sources of these compounds are industrial emissions and discharges although hydrogen sulphide may be present at high levels in areas where there are large amounts of decaying sulphur containing organic material.

Inhalation of carbon disulphide may lead to irritation of the mucous membrane, dizziness and even unconsciousness. The maximum allowable exposure per 8 hours is 20 ppm.

Carbon disulphide is used as a solvent and in the manufacture of rayon and carbon tetrachloride. Thiophene is used as a solvent. Mercaptans are found commonly in bleached Kraft Mill effluents from pulp and paper manufacture. These compounds are extremely toxic to fish, with sub-lethal effects observed below 1 mg/L. Hydrogen sulphide is often associated with organo-sulphur compounds and has similar health effects as carbon disulphide.

Sampling

Aqueous samples should be collected in 1 liter glass bottles with foil lined caps. Solid samples should be collected in pomade jars and air samples should be collected in polyester-protected aluminized sampling bags. Aqueous and solid samples should be kept cool and transferred to the laboratory as soon as possible. Bags should be kept at room temperature and in darkness.

Method

Aqueous and solid samples are analyzed for organo-sulphur compounds and hydrogen sulphide by gas chromatography through head space analysis of the vapour the sealed bottles fitted with "Teflon" septa. Air samples are analyzed by direct injection.

ORGANO-SULPHUR- AND SULPHUR COMPOUNDS												
	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum sample Size	1000 mL	1000 mL		1000 mL	1000 mL	20 grams	20 grams	20 grams	20 grams			Half full bag
Preferred Sampling Container	Glass bottle	Glass bottle		Glass bottle	Glass bottle	Glass bottle	Pomade jar	Pomade jar	Pomade jar			Sampling bag
Preservation Technique	4 ^o C	4 ^o C		4 ^o C	4 ^o C	4 ^o C	4 ^o C	4 ^o C	4 ^o C			Keep in dark place
Operating Range	>1.0	>1.0		>1.0	>1.0	>5.0	>5.0	>5.0	>5.0			>0.5
Limit Detection Criteria	1.0	1.0		1.0	1.0	5.0	5.0	5.0	5.0			0.5
Std. Dev. for in run Duplicates												
Precision	*	*		*	*	*	*	*	*			*
Units	mg/L	mg/L		mg/L	mg/L	µg/g	µg/g	µg/g	µg/g			mg/m ³

* = Replicate analyses should be with 10% of the mean.

OXYGEN - DISSOLVED

An adequate supply of dissolved oxygen (DO) is required for maintaining self-purification processes in natural water systems and waste treatment plants. DO data determined in the field provides a rapid method of assessing the effects of oxidizable wastes on receiving waters, the efficiency of waste treatment during biochemical oxidation and the capacity of a natural body of water for maintaining aquatic life. Dissolved oxygen in water may be derived from atmospheric transfer or photosynthetic processes.

The Water Management Objectives state that at no time should dissolved oxygen concentrations be lower than the values below to protect aquatic life. Any reduction of dissolved oxygen below the levels given will have some effect on the ecosystem. The magnitude of the effect will depend on the severity and duration of the reduction.

Temperature °C	Dissolved Oxygen Concentration mg/L	
	Cold Water Biota	Warm Water Biota
0	8	7
5	7	6
10	6	5
15	6	5
20	5	4
25	5	4

Ideally, DO measurements should be made at the sampling site. The effects of temperature, pressure, bacteriological activity, high organic content and time in transit could significantly change the DO concentration when the sample is transported.

The test measures molecular oxygen as mg/L of solution.

Sampling

Samples should be collected in glass bottles (Prince of Wales) with polyseal cone caps and filled to capacity to ensure that no air bubbles are trapped. Samples should be refrigerated and protected from sunlight to minimize bacteriological and photosynthetic activity.

Method

Samples are usually measured using an oxygen electrode which has a gas permeable membrane. Any gas which passes through the membrane and which has a reduction potential close to that of oxygen may interfere.

Samples may also be analyzed using the Modified Winkler Technique.

Time of Analysis

Approximately 200 dissolved oxygen analyses could be performed per day using the oxygen electrode.

OXYGEN DISSOLVED

Minimum Sample Size

Preferred Sampling Container

Preservation Technique

Operating Range

Limit Detection Criteria

Std. Dev. for in run Duplicates

Precision

Units

Surface Water

Drinking Water

Precip. Sewage

Ind. Wastes

Sludge

Soil & Sediment

Veg'n

Bio Material

Hi-Vol

Dust Fall

Ambient Air

Full bottle

Prince of Wales bottle

4°C Keep in dark

0 to 15

<20% for in run Duplicates

mg/L as O₂

mg/L as O₂

PARTICLE SIZE DISTRIBUTION

The solid phase of soil and sediment is comprised of structurally diverse organic and inorganic particles which may be crystalline or amorphous and which vary greatly in size and shape. The measurement is generally restricted to the inorganic component. Although these particles are generally found as aggregates, they can be separated by chemical and mechanical means.

Many soil textural classification systems are based on particle size measurements, however, the variety of procedures used makes comparisons between classification schemes difficult. Particle size is a significant controlling factor for porosity, permeability, water holding capacity, leaching potential, cation exchange, weathering behaviour and several other parameters. Textural measurements on bottom and suspended sediments may be used to determine relationships between water quality, flora and fauna populations and bottom sediment characteristics. The fractionation of soils and sediments into various size classes can be useful for a correlation of pollutant concentration with texture.

Sampling

Samples should be collected in glass or plastic bottles.

Method

Method A involves sieve analysis to separate the sand fraction. Method B employs a hydrometer to measure changes in density of a suspension in a sedimentation cylinder. Density changes can be related to the settling velocity of the particles as determined by Stokes Law. Method C employs the pipette method in which aliquots are withdrawn from a sedimentation cylinder at a predetermined time. This aliquot is then dried and weighed. This method requires more time and care than method B. Method D involves the use of a Sedimentation balance. This method allows more accurate measurement of the silt fraction than Methods B or C.

Time of Analysis

Method A - 1 day per sample if pretreatment is required.
Method B - 10 samples per 24 hours.
Method C - 10 samples per 24 hours.
Method D - 1 sample per 24 hours.

PARTICLE SIZE
DISTRIBUTION

Method

	A Sieve Analysis	B Hydrometer	C Pipet Method	D Sedimentation Balance
Min. sample size (grams)	20 - 30	10	1	1
Preferred Sampling Container	Pomade jar	Pomade jar	Pomade jar	Pomade jar
Preservation Technique	None	None	None	None
Operating Range	0 to 100	0 to 100	0 to 100	0 to 100
Detection Limit Criteria	-	-	-	-
Precision	-	-	-	-
Units (in each size classification)	%	%	%	%

PETROLEUM HYDROCARBONS

Petroleum hydrocarbons include gasolines, aviation fuels, organic cleaners, thinners, kerosenes, stove oils, home heating fuels and transformer oils. Many of these substances may impart objectionable tastes and odours to domestic water supplies, even at low levels. They are also known to increase the oxygen depletion rate in receiving waters and will hinder oxygen exchange with the atmosphere by forming slicks.

Petroleum hydrocarbons are generally found as a complex mixture of organic compounds. Thus, identifications may be reported as a class or type of material rather than as a specific substance. Decomposition of the sample may alter part of the chromatogram which is used for quantitative purposes. Although the results are considered to be only semi-quantitative they may give an indication of the magnitude of pollution present. Qualitative results are obtained by direct comparison with known standards. The source of the contaminant may be identified by comparing the chromatogram of the extracted pollutant with those of samples collected from storage tanks in the spill area. Chromatograms are generally assessed qualitatively and semi-quantitatively for liquid samples and qualitatively for soil samples. To protect fresh water fish, concentrations should not exceed 180 $\mu\text{L/L}$ for bunker oil, 120 $\mu\text{L/L}$ for used crankcase oil, 45 $\mu\text{L/L}$ for jet aviation fuel and crude oil slicks, 15 $\mu\text{L/L}$ for diesel oil and 9 $\mu\text{L/L}$ for automobile gasoline.

Sampling

Open waters should be sampled at the surface as this is where petroleum hydrocarbons will collect. If closed plumbing systems are to be sampled, taps should **not** be opened for several hours prior to collection so that contaminants may build up. Tap sieves and toilet tanks are frequent areas where contaminants collect. Samples of the suspected contaminant source should be submitted with the contaminated water whenever possible.

One full 32 ounce bottle, clearly labelled "PETROLEUM HYDROCARBONS" should be submitted for this test exclusively. Bottle caps must be aluminum foil or Teflon lined as any rubber or plastic caps may release extraneous organic compounds. Containers must be tightly capped and refrigerated to prevent the loss of volatile components. Solid samples should be stored in pomade jars and kept refrigerated. Petroleum hydrocarbons are susceptible to bacterial attack; therefore immediate delivery to the laboratory is essential.

Method

An extraction is performed using an organic solvent such as n-hexane and analysis is accomplished by gas chromatography.

Time of Analysis

Approximately 5 to 10 petroleum hydrocarbon determinations can be performed per day.

pH

pH is defined as the negative logarithm of the hydrogen ion activity, but in the case of natural water samples, the hydrogen ion activity closely approximates the hydrogen ion concentration in moles per liter. As the pH scale is logarithmic, a change of pH by one unit means a tenfold change in the hydrogen ion concentration. Rivers and lakes which have not been polluted by acid or alkaline wastes range in pH from about 5 (acid peat bogs) to 8.5 (chalk streams and marl lakes). In highly productive waters, photosynthetic activity can cause the overabundance of carbonate ion. This may also result in a natural pH close to 8.5.

Most surface waters are protected from fluctuations in pH by the natural buffering capability of the bicarbonate ion. Pollution by industrial wastes and acid precipitation, however, can upset this equilibrium and produce significant pH changes in the receiving water. A change in pH implies that changes in the alkalinity and acidity are also taking place. pH results are best interpreted in conjunction with the alkalinity and acidity.

pH is a highly perishable parameter because of its dependence on temperature, dissolved gas concentrations, and other chemical reactions. It should be measured in the field whenever possible. A laboratory pH measurement is not necessarily representative of the pH at the time of sampling.

Because both alkaline and acidic waters may cause eye irritation, the pH of the water used for recreational purposes should be within the range of 6.5 to 8.5.

Sampling

Bottles should be filled to capacity so that no air bubbles remain once the samples are capped. For sludges, use wide mouth glass bottles and fill only half way. Do not refrigerate. Immediate delivery to the laboratory is essential. At least 100 mL are required if low level alkalinity or acidity tests are to be performed on this sample. Aqueous samples should be collected in pyrex or polystyrene bottles while soil samples should be collected in pomade jars.

Method

The pH of water samples is measured directly on a stirred aliquot of sample at room temperature. A high quality pH meter is used. The pH of soil and sediment samples is measured on a 0.01 M CaCl_2 or distilled water extract. The sample temperature, degree of electrode immersion and stirring rate should all be as constant as possible.

Time of Analysis

Approximately 100 pH analyses can be performed per day.

pH	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	50 or 100 mL	50 mL	100 mL	50 mL	50 mL	50 mL	10 grams					
Preferred Sampling Container	Pyrex or poly-styrene	Pyrex or poly-styrene	Poly-styrene	Glass bottle	Glass bottle	Glass bottle	Pomade jar					
Preservation Technique	See text	See text	See text	See text	See text	See text	4°C					
Operating Range	4 to 14	6.5 to 9.0	3.5 to 7	7 to 9	7 to 9	7 to 9	2 to 12					
Limit Detection Criteria												
Std. Dev. for in run Duplicates	0.026 0.0315 -	0.023 -	0.022 -	0.0277 -	0.0277 -	0.0277 -	0.0227 -					
Precision												
Units	pH units	pH units	pH units	pH units	pH units	pH units	pH units					

PHENOLICS

Reactive phenolics are compounds having one or more hydroxy groups attached directly to an aromatic ring.

Phenolic compounds are present in the aquatic environment as a result of natural processes such as the decomposition of lignin and degradation of biocides and pesticides, and industrial processes such as the steel, petrochemical and petroleum, pulp and paper and plastics industries.

The formation of chlorophenols such as ortho-chlorophenol, para-chlorophenol, 2,6-dichlorophenol and 2,4-dichlorophenol during chlorination processes may produce taste and odour problems in treated water supplies. In many cases, these chlorinated phenols cannot be detected by the available chemical method. Samples should be collected before chlorination.

High levels of phenolic compounds are objectionable because they may exert a high oxygen demand on a water, may affect aquatic biota by direct toxicity or by imparting objectionable taste and odour to them and may be harmful to humans.

Substituted phenols with alkyl, aryl, nitro, benzoyl, nitroso or aldehyde groups in the para position are generally not measured by the test currently employed. The choice of phenol as the standard for the colorimetric test means that any color produced by the reaction of other phenolics is reported as phenol. Substitution generally reduces recovery and thus the reported value represents the minimum concentration of phenolics present in the sample. The maximum accepted limit for phenolics in drinking water is 1 µg/L. Concentrations of phenols should not exceed 1 µg/L to protect against tainting of edible fish flesh.

Sampling

Samples should be collected in special bottles containing the proper amount of preservative to inhibit bio-degradation and chemical oxidation (Green label). They should be refrigerated and delivered to the laboratory as quickly as possible. (Collect before chlorination).

Method

Aqueous samples are acidified with sulphuric acid and distilled in-line at atmospheric pressure and 120 °C. Analysis is then performed colorimetrically using a 4-aminoantipyrine method which has been adapted to an AutoAnalyzer system. Phenols in solid samples are determined after manual distillation by the Gibb's method.

Time of Analysis

Approximately 100 phenolics determinations can be performed per day by AutoAnalyzer.

PHENOLICS	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	Full bottle	Full bottle		Full bottle	Full bottle		10 grams					
Preferred Sampling Container	See text	See text		See text	See text		Pomade jar					
Preservation Technique	4°C	4°C		4°C	4°C		4°C					
Operating Range	0.3 to 50.0	0.3 to 50.0		0.3 to 50.0	0.3 to 50.0		1 and up					
Limit Detection Criteria	0.58	0.58		0.58	0.58		1					
Std. Dev. for in run Duplicates	0.175 0.744	0.175 0.744		0.175 0.744	0.175 0.744							
Precision												
Units	µg/L	µg/L		µg/L	µg/L		µg/g					

PHENOXY ACID HERBICIDES

Phenoxacetic acids herbicides (2,4-D type) are widely used for selective weed control in crops that are comparatively resistant to these chemicals and for the control of roadside vegetation. These herbicides may also be used for the control of some aquatic plants. Potential problems occur when agricultural run-off waters containing 2,4-D type residues are used to irrigate a crop which is more susceptible to these residues, thus causing serious damage. These materials can also impart severe taste and odour problems to water supplies at low milligram per liter levels. Part of the taste problems arise from the ability of the herbicides to decompose chlorophenols which may be present as impurities in the herbicide.

Although the toxicity of 2,4-D type herbicides to mammals is relatively low, impurities (such as dioxins) which are produced as by-products in the manufacture of these herbicides are extremely toxic. Such impurities may cause birth defects.

The maximum permissible levels in raw waters for 2,4-D, 2,4-DB, 2,4,5-T, 2,4-DP, silvex and MCPA are 100 µg/L. Levels for protection to aquatic life are generally not specified, but 2,4-D and silvex should not exceed 4.0 and 2.4 µg/L respectively. The Environmental Protection Service has designated picloram as high, 2,4-D, 2,4,5-T and silvex as moderate and 2,4-DB and 2,4-DP as slight ecological hazards.

Sampling

Aqueous samples should be collected in solvent cleaned brown glass bottles with foil lined caps. Sampling bottles should be filled to the mark and preserved with 5 mL of concentrated phosphoric acid. Solid samples should be collected in solvent rinsed pomade jars with foil lined caps. All samples should be refrigerated and sent to the laboratory as quickly as possible.

Method

All samples undergo solvent extraction, evaporation, derivatisation, clean-up with Florisil and analysis by electron capture gas chromatography.

Time of Analysis

Approximately 8 samples can be analyzed per day.

PHENOXY ACID HERBICIDES	Surface Water	Drinking Water	Precip. Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	800 mL	800 mL	800 mL	800 mL		10 grams	10 grams	10 grams			
Preferred Sampling Container	Special bottle	Special bottle	Special bottle	Special bottle		Special jar	Special jar	Special jar			
Preservation Technique	5 mL H_3PO_4 $4^\circ C$	5 mL H_3PO_4 $4^\circ C$	5 mL H_3PO_4 $4^\circ C$	5 mL H_3PO_4 $4^\circ C$		$4^\circ C$	$4^\circ C$	Freeze			
Operating ** Range	>0.05	>0.05	>0.05	>0.05		>0.05	>0.05	>0.05			
Limit	0.05 to 0.20	0.05 to 0.20	0.05 to 0.20	0.05 to 0.20		0.05 to 0.02	0.05 to 0.20	0.05 to 0.20			
Detection Criteria											
Std. Dev. for in run Duplicates											
Precision	*	*	*	*	*	*	*	*			
Units	$\mu g/L$	$\mu g/L$	$\mu g/L$	$\mu g/L$	$\mu g/L$	$\mu g/g$	$\mu g/g$	$\mu g/g$			

* = Replicate analyses should be within 10% of the mean.

** = Actual operating range may vary from the detection limits shown to percent levels.

PHOSPHORUS - FILTERED REACTIVE (ORTHOPHOSPHATE)

The filtered reactive phosphorus content of a aqueous sample is that fraction which passes through a filter and reacts with the reagents present to produce a positive colorimetric response. It is possible that some organic and particulate forms of phosphate can react similarly to dissolved orthophosphate under the analytical test conditions. The above terminology should remove the implication that the test measures only orthophosphate forms which are uncombined and in solution.

Phosphorus is an essential element for all life forms. Artificial inputs of phosphorus via wastewater and land run-off play a significant role in promoting an overabundance of algae and aquatic plants which may impair water quality.

Phosphates are used in some water treatment methods, in commercial cleaning preparations, to treat boiler waters, and as fertilizer.

The usefulness of filtered reactive phosphorus results is questionable because of its reactivity. Results must be interpreted with full allowance for perishability, both in situ and in the sample following collection.

Sampling

Use only brand new glass or polystyrene containers when sampling for filtered reactive phosphorus. Re-washed containers are likely to remain contaminated with a residual amount of phosphorus, regardless of how thoroughly they are washed. Filtered reactive phosphorus is readily converted to other phosphorus forms biologically so samples must be refrigerated or frozen immediately after collection and delivered to the laboratory as soon as possible.

Method

Samples are filtered and analyzed colorimetrically using an ammonium molybdate-ascorbic acid method which has been adapted to an AutoAnalyzer system. High levels of iron (III), hexavalent chromium, arsenic (V), stannous ion, and some colored compounds are known to interfere. Results are reported as mg/L of phosphorus.

Time of Analysis

Approximately 200 analyses may be performed per day. Other soluble nutrients such as nitrite, nitrate and ammonia may be determined at the same time.

PHOSPHORUS FILTERED REACTIVE (ORTHOPHOSPHATE)	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	75 mL	75 mL	mL	75 mL	75 mL							
Preferred Sampling Container	Glass or poly- styrene	Glass bottle		Glass bottle	Glass bottle							
Preservation Technique	Freeze or 4 °C	Freeze or 4 °C		4 °C	4 °C							
Operating Range	0.004 to 0.100	0.04 to 4.00		0.04 to 4.00	0.04 to 4.00							
Limit Detection Criteria	0.004	0.090		0.090	0.090							
Std. Dev. for in run Duplicates	<20% 0.0011 0.0015 0.0010	0.0275 0.0435 0.0636		0.0275 0.0435 0.0636	0.0375 0.0435 0.0636							
Precision												
Units	mg/L Phosphorus	mg/L Phosphorus		mg/L Phosphorus	mg/L Phosphorus							

PHOSPHORUS - FILTERED TOTAL

Phosphorus can exist in natural waters and wastewaters in a number of forms, which include orthophosphates, pyrophosphates, metaphosphates, polyphosphates and organically bound phosphates. The filtered total phosphorus test differs from the total phosphorus test in that the digestion procedure is not as severe. While both methods promote hydrolysis of the various forms of phosphorus to orthophosphate, the filtered total phosphorus digestion does not release organically bound phosphorus to any great extent. Results from the filtered total phosphorus test are usually used in primary productivity studies.

Sampling

Acid washed Pyrex or Kimax tubes with Teflon liners should be obtained from Central Stores. Samples must be filtered in the field using 0.45 μm membrane filters. Consult laboratory staff for acceptable types of filters. Tubes must be filled above the mark.

Method

Samples are pretreated by autoclave digestion in a sulphuric acid - potassium persulphate medium for one hour at 121 $^{\circ}\text{C}$. Analysis is performed colorimetrically using an ammonium molybdate - ascorbic acid method which has been adapted to an AutoAnalyzer system. High levels of arsenate, ferric ion and silicates interfere. Results are reported in $\mu\text{g/L}$ of phosphorus.

Time of Analysis

Approximately 150 samples can be analyzed per day.

PHOSPHORUS
 FILTERED
 TOTAL

Surface
 Water

Drinking
 Water

Precip.

Sewage

Ind.
 Wastes

Sludge

Soil &
 Sediment

Vegh

Bio
 Material

Dust
 Fall

Hi-Vol

Ambient
 Air

Minimum
 Sample
 Size

Full
 tube

Preferred
 Sampling
 Container

Pyrex
 tube

Preservation
 Technique

None

Operating
 Range

<20

Limit
 Detection
 Criteria

Std.
 Dev.
 for
 in run
 Duplicates

<20%
 20-50%
 50-100%

Precision

Units

$\mu\text{g/L}$
 Phosphorus

PHOSPHORUS IN SOIL, SEDIMENT AND VEGETATION

This outline includes two types of tests: Extractable Phosphorus and Total Phosphorus by dissolution.

Phosphorus occurs in nature in a variety of organic and inorganic forms. There is a continual process of uptake of inorganic forms, conversion to organically bound phosphorus and decomposition of these organic forms to oxidized inorganic forms.

Phosphorus is derived from leaching and weathering of igneous rocks and phosphorus minerals such as apatite, domestic sewage and fertilizers. Phosphorus in acidic soil and sediment is often associated with iron and aluminum while it is associated with calcium in alkaline materials. While most forms of phosphorus are biologically available, apatite phosphorus is not.

The extractable phosphorus tests for soil and sediment use one or a sequence of extractions to determine different forms of phosphorus, whereas the total phosphorus by dissolution uses only a one step digestion to dissolve the total amount of phosphorus present.

Sampling

Samples for extractable phosphorus should be collected in pomade jars, refrigerated and air-dried. Samples for total phosphorus can be air or oven dried.

Method

Extractable phosphorus

Samples are extracted sequentially with sodium hydroxide (for iron and aluminum phosphates), a sodium citrate-bicarbonate-dithionate solution (for occluded phosphates) and hydrochloric acid for calcium phosphate. Phosphorus in the various solutions is analyzed colorimetrically using an ammonium molybdate-ascorbic acid manual method. Sodium bicarbonate is used as an extractant for "available" phosphorus in sewage sludge amended soils.

Total phosphorus by dissolution

All phosphorus is converted to the inorganic form by digestion in hot sulphuric acid with the addition of potassium persulphate. After elimination of interfering iron in soil and sediment digestates by precipitation and filtration, phosphorus is measured colorimetrically using an ammonium molybdate-ascorbic acid AutoAnalyzer method.

PHOSPHORUS IN SOIL, SEDIMENTS & VEGETATION EXTRACTABLE PHOSPHORUS	Soil & Sediment	PHOSPHORUS IN SOILS, SEDIMENTS & VEGETATION TOTAL PHOSPHORUS BY DISSOLUTION	Soil & Sediment
Minimum Sample Size	0.1 grams		1 grams
Preferred Sampling Container	Pomade jar		Pomade jar
Preservation Technique	4 °C		4 °C
Operating Range	0.1 to 1.0		0.01 to 1.6
Limit Detection Criteria	0.1		0.01
Std. Dev. for in run Duplicates	<20% 20-50% 50-100%		
Precision	Consult staff		0.049 at 0.7
Units	mg/g		mg/g

PHOSPHORUS - TOTAL

Phosphorus occurs in natural waters and wastewaters in several different combinations, which include orthophosphates, pyrophosphates, metaphosphates, polyphosphates and organically bound phosphates. Phosphorus is an essential element for all life forms. As all of the above compounds may eventually be used in metabolic processes, determination of the total phosphorus concentration is more relevant than the measurement of individual phosphorus compounds.

Phosphates are used in the manufacture of fertilizers as well as detergents. Polyphosphates are used in some public water supplies and industrial boiler systems as scale and corrosion inhibitors. Artificial inputs of phosphorus via wastewater and land run-off play a significant role in promoting the over-abundance of algae and aquatic plants which impair water quality. Phosphorus results are used in assessing a water's potential for biological productivity as well as the efficiency of nutrient removal facilities at waste treatment plants. To avoid nuisance levels of algae in lakes, average total phosphorus concentrations for ice free periods should not exceed 20 $\mu\text{g/L}$. To protect against aesthetic deterioration, total phosphorus levels should not exceed 10 $\mu\text{g/L}$ during ice free periods.

Sampling

Contamination is a significant problem in determining total phosphorus concentrations. Re-washed bottles may contain residues of phosphorus, hence single-use glass containers or plastic are preferred. If using a re-washed container is unavoidable, it must be cleaned with 10% hydrochloric acid and thoroughly rinsed with distilled water prior to field use. Soil samples should be collected in pomade jars. Vegetation samples should be collected in plastic bags and refrigerated. Refrigeration or freezing is required only if other nutrient parameters are to be determined on the same sample.

Method

Aqueous samples are pre-treated by digestion in a sulphuric acid, mercuric oxide and potassium sulphate medium and analysis is accomplished colorimetrically using an ammonium molybdate - ascorbic acid technique which has been adapted to an AutoAnalyzer system. High levels of iron(III), arsenate, and hexavalent chromium are known to interfere. Interferences by colored compounds should be removed by the digestion procedure. Results are reported as mg/L of phosphorus.

Time of Analysis

Approximately 200 samples may be analyzed per day. Total Kjeldahl nitrogen is performed simultaneously.

PHTHALIC ACID ESTERS

The aromatic dicarboxylic esters such as the phthalic acid esters are important industrial chemicals. They are used as plasticizers for PVC, and polyvinyl acetate, polystyrene, cellulose nitrate, cellulose acetate and many others. Compounds which can be measured include dimethyl-, diethyl-, dipropyl-, dibutyl-, dioctyl-, diethyl-, hexyl-, and dinonyl-phthalate.

Phthalic acid esters have been found in water, sediments, fish and vegetation. The phthalic acid esters are lipid soluble and can move from plastic packaging into foods. They are generally non-persistent in water and aerobic sediments but they may persist in anaerobic sediments. As a result, bottom dwelling fauna may be exposed to high levels of phthalic acid esters.

Phthalic acid esters may contribute to shock lung disease, a condition characterized by an impeded circulation of blood in the lungs. Phthalic acid esters may be accumulated by aquatic life at levels several thousand times higher than ambient levels.

Objectives for a few phthalates based on toxicity data are given below:

Dibutylphthalate	4.0 $\mu\text{g/L}$
Diethylhexylphthalate	0.6 $\mu\text{g/L}$
other phthalates	0.2 $\mu\text{g/L}$

Sampling

Surface and drinking water, industrial and domestic waste samples should all be collected in 1 liter glass bottles with foil lined caps. Samples should be kept cool and should be transported to the laboratory as quickly as possible.

Method

The samples are analyzed by gas chromatography following extraction with an appropriate solvent.

PHTHALIC ACID ESTERS	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	1000 mL	1000 mL		1000 mL	1000 mL							
Preferred Sampling Container	Glass bottle	Glass bottle		Glass bottle	Glass bottle							
Preservation Technique	4°C	4°C		4°C	4°C							
Operating Range	>2	>2		>2	>2							
Limit Detection Criteria	2 - 5	2 - 5		2 - 5	2 - 5							
Std. Dev. for in run Duplicates												
Precision	*	*		*	*							
Units	μ g/L	μ g/L		μ g/L	μ g/L							

* = Replicate analyses should be within 10% of mean.

POLYNUCLEAR AROMATIC HYDROCARBONS (PAH)

Polynuclear aromatic hydrocarbons are extensively studied in polluted air and food products. Several members of this chemical class have been shown to be carcinogenic to animals and several are suspected carcinogens for man. Besides benzo(a)pyrene, at least 11 carcinogenic PAH's have been identified in urban air. At present no criteria have been established for these compounds.

PAH's are formed during heating, burning, incineration and other combustion processes of many types of carbonaceous materials. They are also associated with coal, oil and gas burning power plants, steel and petrochemical industries. Generally, PAH's in nature are produced by combustion of organic matter under conditions of insufficient amounts of oxygen and/or low combustion temperatures.

In the presence of air, light and/or heat, some PAH's are reported to undergo chemical changes such as dimerization, photodegradation and oxidation particularly when dissolved in solvents or adsorbed on active surfaces. PAH compounds detected include fluoranthene, perylene, benzo(k)fluoranthene, benzo(a)pyrene, benzo(ghi)perylene and ortho-phenylene pyrene. These compounds may be concentrated in fish from 100 to 1000 times the ambient concentration.

Sampling

Aqueous samples should be collected in 1 liter glass bottles with foil lined caps or in impingers. Solid samples should be collected in pomade jars. Air samples (total suspended particulate) should be collected on glass fibre Hi-Vol filters which are kept in envelopes protected from light, heat and humidity prior to analysis. Aqueous and solid samples should be kept cool and transported to the laboratory as quickly as possible.

Method

Aqueous samples are preconcentrated with an organic solvent and analyzed by high performance liquid chromatography (HPLC) and a fluorescence detector. Solid samples are washed with an organic solvent and analyzed by the same method. Air samples are analyzed after extraction from the filters with cyclohexane.

POLYNUCLEAR AROMATIC HYDROCARBONS												
	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	1000 mL	1000 mL		1000 mL	1000 mL	20 grams	20 grams	20 grams	20 grams		1/20 glass fibre filter	
Preferred Sampling Container	Glass bottle	Glass bottle		Glass bottle	Glass bottle	Glass bottle	Pomade jar	Pomade jar	Pomade jar			
Preservation Technique	4 ^o C	4 ^o C		4 ^o C	4 ^o C	4 ^o C	4 ^o C	4 ^o C	4 ^o C		None	
Operating Range	>0.1	>0.1		>0.1	>0.1	>1.0	>1.0	>1.0	>1.0		>0.01	
Limit Detection Criteria	0.1	0.1		0.1	0.1	1.0	1.0	1.0	1.0		0.01	
Std. Dev. for in run Duplicates												
Precision	*	*		*	*	*	*	*	*		*	
Units	ng/L	ng/L		ng/L	ng/L	ng/g	ng/g	ng/g	ng/g		μg/m ³	

* = Replicate analyses should be within 10% of the mean.

POTASSIUM

Potassium ranks seventh in the natural order of elemental abundance. It is highly soluble and present at trace levels in all natural surface water, but very seldom exceeds 10 mg/L. Brackish or saline ground water, however, may contain potassium in excess of 100 mg/L. Potassium is generally found in the presence of sodium and only in rare instances will it ever exceed the sodium concentration. The sodium:potassium ratio in natural waters generally ranges from 2:1 to 3:1 with the proportion of sodium increasing with increasing concentrations. When potassium is in greater abundance than sodium, an artificial source is likely.

Potassium is an essential nutrient for all life forms, especially plants, and is therefore a major component of commercial fertilizers. Despite the substantial amounts of potassium dispersed over the land as fertilizer, it seldom appears in any great quantity in the water system. Many soils have the ability to fix potassium and prevent its migration through leaching. It is non-toxic and represents no known health hazard unless at extremely high levels. No limits have been specified for the maximum acceptable concentration of potassium in domestic water supplies.

Sampling

Under alkaline conditions, glass containers are attacked releasing potassium ions to the sample. Pyrex is more resistant than soft glass, but plastic containers are preferred. Glass containers are only adequate when neutral pH conditions are known to exist. Soil samples should be collected in pomade jars. Vegetation samples should be collected in perforated plastic bags and kept refrigerated. Fish samples should be placed in plastic bags and frozen.

Method

River, precipitation and domestic water samples are analyzed using an atomic absorption technique which has been modified to an automated system. Cesium chloride is added to minimize partial ionization of potassium ions in the flame.

Soil and vegetation samples are analyzed using atomic absorption after ashing and solution in hydrochloric acid.

Potassium in vegetation samples is also measured by X-ray fluorescence. Results are reported as % dry weight of the sample (See Matrix elements in Vegetation).

The Thunder Bay laboratory uses flame emission to analyze for potassium.

Time of Analysis

Approximately 150 potassium analyses may be performed per day on aqueous samples and 50 per day on solid samples.

POTASSIUM	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	50 mL	50 mL	50 mL	50 mL	50 mL		2 grams	2 grams	50 grams			
Preferred Sampling Container	Plastic or pyrex glass	Plastic or pyrex glass	Poly-styrene	Pyrex glass	Pyrex glass		Pomade jar	Plastic bag	Plastic bag			
Preservation Technique	None	None	4°C	None	None		None	4°C	Freeze			
Operating Range	0.07 to 5.00	0.1 to 20.0	0.02 to 2.00	0.1 to 20.0	0.1 to 20.0		5 to 200	5 to 200	*			
Limit Detection Criteria	0.132	0.122	0.143	0.122	0.122		5	5				
Std. Dev. for in run Duplicates	0.040 0.050 0.061	0.037 0.080 0.110	0.013 - -	0.037 0.080 0.110	0.37 0.080 0.110		8 at 100	8 at 100				
Precision												
Units	mg/L	mg/L	mg/L	mg/L	mg/L		µg/g	µg/g	µg/g			

* = Test available upon request.

RADIONUCLIDES

Radionuclides may be released from nuclear plants, uranium mining and processing plants, hospitals and by seepage from radioactive wastes.

Three radionuclides which are measured by this laboratory are phosphorus-32, tritium and carbon-14.

Tritium can be used for dating ground waters and tracing their movement. Phosphorus-32 and Carbon-14 are used for studying primary productivity and elemental uptake.

Radiochemical techniques can be applied to many diverse research problems and sample matrices. Consultation with laboratory staff is required prior to sampling and submission.

Sampling

Sampling should be done in consultation with laboratory staff. They will recommend the proper containers and techniques.

Method

Samples are analyzed by two techniques: liquid scintillation and alpha spectrometry. Liquid scintillation is an indirect but sensitive technique for the analysis of beta emitting radionuclides. Alpha spectrometry involves counting alpha particles (^4He) using a surface barrier detector/multichannel analyzer combination.

[illegible]

SELENIUM

Selenium is naturally found in the form of metallic selenides, selenites, and selenates, and also as organic compounds derived from decayed plant material. Selenium is likely present in natural waters as the selenate anion (SeO_4^{2-}) or as organic selenium compounds which have been discharged in industrial effluents. The elemental form is insoluble in water but it may be transported by being adsorbed onto particulate matter. Sediment material seems to act as sinks or reservoirs for selenium. While water which has seeped through coal and shale deposits may contain up to 0.3 mg/L most surface waters contain less than 0.001 mg/L. Its common industrial applications include the manufacture of rectifiers, photoelectric cells, red colored glass, pigments, paints, dyes and insecticides, as well as in the vulcanization of rubber as a supplement to sulphur.

Deficiency of selenium in the grass eaten by livestock may lead to white muscle disease. Selenium poisoning in mammals may be of the acute type, called "blind staggers", or the chronic type, called "alkali disease". In humans, acute toxicity may be characterized by nervousness, vomiting, cough, convulsions, abdominal pains, diarrhea and respiratory failure. Chronic effects include weakness, visual impairment, damage to the heart and liver, tooth decay, red tainting of fingers, teeth and hair, depression and irritation of nose and throat. Claims of carcinogenic potential have yet to be proven.

Selenium may decrease the toxicity of cadmium and mercury.

The 96 hour LC_{50} values for zebra fish larvae indicate that selenates are less toxic than selenite salts by about a factor of 4. A lethal threshold value of 2.5 mg/L has been found for Daphnia sp. and Scenedesmus sp.

The maximum acceptable concentration of selenium for domestic water supplies in Ontario is 0.01 mg/L. Concentrations in an unfiltered sample should not exceed 0.1 mg/L to protect aquatic life. Fish with levels of 3 $\mu\text{g/g}$ wet weight, should not be eaten often. Livestock waters should not contain more than 0.05 mg/L while irrigation waters should not contain more than 0.02 mg/L.

Sampling

Glass containers may be used for all samples. Vegetation samples should be collected in plastic bags and refrigerated. Fish samples should be placed in plastic bags and frozen.

Method

Samples are analyzed using flameless atomic absorption.

Time of Analysis

Approximately 50 samples can be analyzed per day.

SELENIUM	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	50 mL	50 mL		25 mL	25 mL	2 mL	0.2 grams	0.2 grams	50 grams			
Preferred Sampling Container	Glass bottle	Glass bottle		Glass bottle	Glass bottle	Glass bottle	Pomade jar	Plastic bag	Plastic bag			
Preservation Technique	None	None		None	None	None	None	4°C	Freeze			
Operating Range	0.001 to 0.20	0.001 to 0.20		0.001 to 0.5	0.001 to 0.5	0.025 to 0.5	0.025 to 2.5	0.025 to 2.5	-			
Limit Detection Criteria	0.001	0.001		0.001	0.001	0.025	0.025	0.025	-			
Std. Dev. for in run Duplicates												
Precision	0.001 at 0.02	0.001 at 0.02		0.005 at 0.100	0.002 at 0.020	0.05 at 0.50	0.05 at 0.50	0.05 at 1.0	-			
Units	mg/L	mg/L		mg/L	mg/L	mg/L	µg/g	µg/g	µg/g			

Std. Dev. for in run Duplicates

SILICON AND SILICATES - REACTIVE

The element silicon ranks second to oxygen in natural abundance in the earth's crust and is present as silica or silicates in sand or quartz. In natural water silicates are generally found as monomers, polymeric chains and cyclic anions in both soluble and colloidal forms. Surface water levels are usually under 5 mg/L but higher levels are not uncommon, especially in ground waters.

Silica and silicates are widely used in industry for the manufacture of glass, ceramics, abrasives and enamels and have been used in domestic water treatment as coagulants, corrosion inhibitors, and sequestering agents for iron. Their presence in boiler feed waters causes deposits of silica on steam-turbine blades, necessitating removal to concentrations as low as 0.1 mg/L in high pressure system.

Silicates are essential to the growth of diatoms as well as many other aquatic organisms. No adverse physiological effects are known at concentrations generally found in natural or treated waters. Therefore, no maximum concentration has been specified for its presence in domestic water supplies.

Sampling

Plastic bottles must be used for aqueous samples requiring silicate analysis. Glass bottles should not be used since silicon is a major constituent of glass. Analyses should be completed as quickly as possible since biological activity can reduce the dissolved silicate concentrations in enriched natural samples. Vegetation samples should be collected in perforated plastic bags and kept refrigerated.

Method

Water samples are analyzed directly using a colorimetric molybdate method which has been adapted to an AutoAnalyzer system. This method measures silicon which is present in the sample as the reactive silicate ion, plus any form of silicon which can be hydrolyzed to this ion under the conditions of the test. The amount of acid and other hydrolyses is unknown but thought to be small. Silica in the soluble colloidal and insoluble polymer forms generally won't react with the molybdic acid. High concentrations of iron, tannins, lignins, sulphides and phosphates may interfere, however, many of these can be overcome. Highly colored or turbid samples may have some interference. Results are reported as mg/L of silicon.

Vegetation samples are analyzed by X-ray fluorescence. Results are reported as % of dry weight. (See Matrix elements in Vegetation).

Time of Analysis

Approximately 200 silicate analyses can be performed per day.

SILICON AND SILICATES REACTIVE

	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	50 mL	50 mL			50 mL							
Preferred Sampling Container	Poly- ethylene bottle	Poly- ethylene bottle			Poly- ethylene bottle							
Preservation Technique	None	None			None							
Operating Range	0.09 to 5.00	0.09 to 5.00			0.09 to 5.00							
Limit Detection	0.174	0.174			0.174							
Detection Criteria	0.087	0.087			0.087							
Std. Dev. for in run Duplicates	<20% 20-50% 50-100%	0.053 0.054 0.066			0.053 0.054 0.066							
Precision												
Units	mg/L Si	mg/L Si			mg/L Si							

SILVER

Silver is found in nature as the native metal, but its chief source is the mineral argentite. Besides being relatively rare, most of its compounds are insoluble; therefore it is highly unlikely that silver would appear in a domestic water supply unless introduced by artificial means. Weathering of argenite and lead-zinc ores containing silver may release silver to the environment. Freshwater concentrations seldom exceed 0.04 mg/L. It is commonly used in the manufacture of silver utensils, jewellery, and solders as well as in the electroplating, photographic, and food processing industries. Other sources include wastes from silver mines, industrial electronics plants and cloud seeding.

Exposure to silver in industry is usually manifested as a condition referred to an argyria; common symptoms include a permanent blue-gray discoloration of the skin and eyes. Although excessive exposure to silver may result in kidney, liver, or spleen damage, no recognizable disturbance of health need accompany this condition. Silver is accumulated in the skin indefinitely.

The maximum acceptable concentration of silver in domestic water supplies is 0.05 mg/L. This limit is used to restrict the use of silver compounds as disinfectants and is indirectly based on public health considerations. Colloidal elemental silver in water is highly toxic and may result in blindness or death. Concentrations of silver in an unfiltered sample should not exceed 0.1 µg/L to protect aquatic life.

Sampling

Samples should be analyzed as soon as possible after collection. Silver compounds are easily adsorbed on the walls of containers.

Special acid washed plastic containers are required for trace level determinations in domestic water and surface water samples. Glass containers are required for domestic waste and industrial waste samples. Add 20 drops of HNO_3 per liter. Soil samples should be collected in pomade jars.

Method

Samples are digested with an appropriate acid mixture and analyzed by atomic absorption.

Time of Analysis

Approximately 50 silver samples may be analyzed per day.

SILVER	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	400 mL	400 mL		50 mL	100 mL	10 mL	2 grams					
Preferred Sampling Container	Plastic bottle Acid washed	Plastic bottle Acid washed		Glass bottle	Glass bottle	Glass bottle	Pomade jar					
Preservation Technique	HNO ₃ pH <2	HNO ₃ pH <2		HNO ₃ pH <2	HNO ₃ pH <2	None	None					
Operating Range	0.0005 to 0.100	0.0005 to 0.100		0.01 to 2.0	0.005 to 1.0	0.05 to 10.0	*					
Limit Detection Criteria	0.0005	0.0005		0.01	0.005	0.05						
Std. Dev. for in run Duplicates												
Precision	0.01 at 0.50	0.01 at 0.50		0.02 at 0.40	0.02 at 0.40	0.02 at 0.40						
Units	mg/L	mg/L		mg/L	mg/L	mg/L	μg/g					

* = Test available upon request.

SODIUM

Sodium is the sixth most abundant element on earth, comprising about 2.6% of the earth's crust, and is found in most natural waters. Weathering of salt deposits is the principal source of sodium in the aquatic environment. Concentration levels may vary from less than 1 mg/L to several thousand mg/L in some briny groundwaters. Sodium salts are used extensively in the manufacturing of chemical products as well as in commercial water softening devices which employ the sodium ion-exchange process. It is abundant in most industrial effluents. Waters that receive surface drainage from roads during the winter season can contain high levels of sodium if sodium chloride is used to control road ice.

Soils high in sodium are generally characterized by low infiltration and permeability rates and are therefore unsuitable for farming.

Sodium is an essential element for all life forms and is generally considered non-toxic. Patients with high blood pressure are often advised to consume water with less than 50 mg/L of sodium. Waters, softened by ion exchange processes may contain sodium in excess of this amount.

Since sodium is generally considered to be non-toxic, no drinking water criteria have been established.

Sampling

Under alkaline conditions, glass containers are attacked releasing sodium ions to the sample. Pyrex is more resistant than soft glass, but plastic containers are preferred. Glass containers should only be used for sampling when neutral pH conditions are known to exist. Vegetation samples should be collected in plastic bags and refrigerated.

Method

Samples are analyzed by an atomic absorption technique which has been modified to an automated system. Potassium or cesium chloride is used to minimize the partial ionization of sodium atoms in the flame.

Time of Analysis

Approximately 150 sodium analyses may be performed per day.

SODIUM	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	50 mL	50 mL	50 mL	50 mL	50 mL	10 mL	2 grams	2 grams		200 mL		
Preferred Sampling Container	Plastic or pyrex glass	Plastic or pyrex glass	Poly-styrene	Pyrex glass	Pyrex glass	Pyrex glass	Pomade Jar	Plastic bag		Sampling jar		
Preservation Technique	None	None	4°C	None	None	None	None	4°C		None		
Operating Range	0.1 to 10.0	0.3 to 100	0.02 to 1.00	0.3 to 100	0.3 to 100	*	5 to 1000	2 to 300		0.06 to 1.0		
Limit Detection Criteria	0.230	0.63	0.047	0.63	0.63		3	2		0.06		
Std. Dev. for in run Duplicates	<20% 0.070 0.076 0.090	0.19 0.32 0.46	0.0143 0.027 0.030	0.19 0.32 0.46	0.19 0.32 0.46							
Precision								5 at 100		-		
Units	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	µg/g	µg/g		µg/m ² / 30 days		

* = Test available upon request.

SOLIDS - DISSOLVED

Dissolved solids refers to the quantity of material that remains as a residue after the water is removed from a filtered portion of sample by evaporation. The test does not determine specific chemical substances but only a general class of substances which have a similar response under the same test conditions. "Total solids" may be found by taking the sum of the suspended and dissolved solids.

Dissolved solids may be determined in three different ways. Conductivity is the method of choice because it is the most precise and economical. Southern Ontario lakes and rivers free of industrial wastes have a dissolved solids content equal to their conductivity times 0.65 ± 0.10 . Some caution should be exercised in interpreting the results since the calculation methods based on either the conductivity or the summation of mineral components consider only the "ionic" dissolved solids.

Dissolved solids must not be added to increase the ambient concentrations by more than 1/3 of the natural concentration to protect aquatic life. The added solids should not significantly alter the overall ionic balance of the receiving waters. Drinking waters should not contain more than 500 mg/L of dissolved solids.

Sampling

Samples should be collected in glass bottles and should be refrigerated.

Method

Dissolved solids are determined:

Gravimetrically on all industrial waste samples as well as any other type of sample where the conductivity is greater than 450 micromhos/cm.

From the conductivity whenever the conductivity is less than 450 micromhos/cm.

From the sum of constituents by special request Ca, Mg, Na, K, Cl, SO_4 , NO_3 , and alkalinity must all be determined on a particular samples in order to apply this technique. This calculation will be performed only on special request.

Time of Analysis

Two days are required to complete the gravimetric analysis. Samples are set up the first day and the weighing operation is performed the second day. Approximately 75 samples may be set up per day.

NOTE:

Total solids will be substituted for dissolved solids only when filtration becomes impractical, e.g. raw sludges, digested sludges, mixed liquors, etc.

SOLIDS DISSOLVED	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	75 mL	75 mL		75 mL	75 mL							
Preferred Sampling Container	Glass bottle	Glass bottle		Glass bottle	Glass bottle							
Preservation Technique	4°C	4°C		4°C	4°C							
Operating Range	<1000	<5000		<5000	<5000							
Limit Detection Criteria												
Std. Dev. for in run Duplicates	<20% 20-50% 50-100%	13.5 29.6 37.8		13.5 29.6 37.8	13.5 29.6 37.8							
Precision												
Units	mg/L	mg/L		mg/L	mg/L							

SOLIDS - IGNITED

The ignited solids test determines both the quantity of material that remains as a residue and the quantity of material that is lost when either the total, suspended, or dissolved solids residue is ignited at 600 °C for one hour. The test does not determine specific substances but only a general class of substances which have a similar response under the same test conditions.

The loss on ignition is due to oxidation of carbon and hydrogen, escape of water of hydration, volatilization of inorganic ammonium salts, decomposition of magnesium carbonate, and loss of any other materials which are volatile or decomposable below 600 °C. Thus, the loss on ignition generally overestimates the organic content. The actual organic content is lower by the amount of water, inorganic salts, etc., released. In the case of clean natural waters, the loss on ignition is due almost entirely to **non-carbonaceous** material; therefore caution should be exercised in interpreting the results on such samples.

Ignited solids results are most useful in the characterization of sewage sludges where it is desirable to estimate organic composition. It is one of the major tests used in assessing the efficiency of sludge digestion.

Any inaccuracies associated with the total, suspended, and dissolved solids determinations are also an integral part of the ignited solids results. Additional errors resulting from mechanical losses due to decrepitation are also a possibility.

Sampling

Samples should be collected in glass bottles and should be refrigerated.

Method

Analysis is accomplished gravimetrically by taking the total, suspended, or dissolved solids residue and igniting it at 600 °C for one hour.

Time of Analysis

Approximately 75 ignited solids determinations may be completed per day.

SOLIDS IGNITED - SUSPENDED	Surface Water	Drinking Water	Sewage	Ind. Wastes	SOLIDS IGNITED - TOTAL	Surface Water	Drinking Water	Sewage	Ind. Wastes	Sludge
Minimum Sample Size	600 mL	600 mL	600 mL	600 mL		75 mL	75 mL	75 mL	75 mL	50 mL
Preferred Sampling Container	Glass bottle	Glass bottle	Glass bottle	Glass bottle		Glass bottle	Glass bottle	Glass bottle	Glass bottle	Glass bottle
Preservation Technique	4°C	4°C	4°C	4°C		4°C	4°C	4°C	4°C	4°C
Operating Range	<1000	<1000	<1000	<1000		<50,000	<50,000	<50,000	<50,000	<50,000
Limit Detection Criteria										
Std. Dev. for in run Duplicates	<20% 20-50% 50-100%	10.14 44.7 144	10.15 44.7 144	10.14 44.7 144	10.14 44.7 144	19.6 109 354	19.6 109 354	19.6 109 354	19.6 109 354	19.6 109 354
Precision										
Units	mg/L	mg/L	mg/L	mg/L		mg/L	mg/L	mg/L	mg/L	mg/L

SOLIDS - SUSPENDED

Suspended solids refers to the quantity of material that may be removed from a water sample by filtration. The test does not determine specific chemical substances but only a general class of substances which have a similar response under the same test conditions.

The measurement of suspended solids is often improperly used in assessing water clarity. Clarity is a function of a water's optical properties and rarely relates to the quantity of suspended material present. Turbidity is a much better measurement of water clarity than suspended solids.

Suspended solids data is useful in evaluating the strength of domestic wastewater and in assessing the efficiency of waste treatment procedures. It is important in calculating loadings to aeration tanks, digesters, lagoons, and sedimentation tanks.

Acquiring a representative aliquot for analysis is the major source of analytical variability in the suspended solids measurement. Results on samples which contain large particles or more than 0.2% suspended material by weight are questionable because of rapid settling. Since there is considerable analytical variability in the results when the suspended solids concentration is less than 10 mg/L, samplers are urged to substitute a turbidity measurement as often as possible.

Sampling

Samples should be collected in glass bottles and should be refrigerated.

A non-routine modification has been developed to extend the detection limit down to 0.2 mg/L. Low level suspended solids determinations are useful in special studies which involve loadings due to land drainage. Laboratory staff should be consulted if low level measurements are required.

Method

Analysis is accomplished by filtering a known volume of sample through a pre-weighed filter paper (Reeve Angel 934 AH). The filter is then dried to constant weight at 103° - 106°C. Results are reported as mg/L of suspended solids.

Time of Analysis

Two days are required to complete the analysis. Samples are filtered the first day and the weighing operation is performed the second day. Approximately 125 samples may be set up per day.

SOLIDS SUSPENDED	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Vegh	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	600 mL	600 mL		600 mL	600 mL							
Preferred Sampling Container	Glass bottle	Glass bottle		Glass bottle	Glass bottle							
Preservation Technique	4°C	4°C		4°C	4°C							
Operating Range	0.5 to 10	<500		<500	<500							
Limit Detection Criteria	0.984	13.1		13.1	13.1							
	0.492	6.53		6.53	6.53							
Std. Dev. for in run Duplicates	<20% 0.299 0.364 0.481	3.97 4.71 7.05		3.97 4.71 7.05	3.97 4.71 7.05							
Precision												
Units	mg/L	mg/L		mg/L	mg/L							

SOLIDS - TOTAL

Total solids refers to the quantity of material that remains as a residue after the water is removed from a sample after evaporation at $103 \pm 2^{\circ}\text{C}$. This test does not determine specific chemical substances, only a general class which have a similar response under the same test conditions.

Total solids are generally estimated from the sum of the suspended and dissolved solids. Gravimetric analyses are normally only performed on samples, such as sludges, which are very difficult to filter.

Sampling

Samples should be collected in glass bottles and should be refrigerated.

Method

Analysis may be performed gravimetrically by evaporating an aliquot of sample overnight at $103 \pm 2^{\circ}\text{C}$. Results may also be obtained from the sum of the suspended and dissolved solids fractions.

Time of Analysis

Two days are required to complete gravimetric analysis. Samples are set up the first day and weighed the second day. Approximately 75 samples may be set up per day.

SOLIDS TOTAL	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Vegh	Bio Material	Dust Fall	Hi-Vol	Ambient Air
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Minimum Sample Size	75 mL	75 mL	75 mL	75 mL	75 mL	50 mL						
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Preferred Sampling Container	Glass bottle	Glass bottle	Glass bottle	Glass bottle	Glass bottle	Glass bottle						
------------------------------------	-----------------	-----------------	-----------------	-----------------	-----------------	-----------------	--	--	--	--	--	--

Preservation Technique	4°C	4°C	4°C	4°C	4°C	4°C						
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Operating Range					<400,000	<400,000						
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Limit Detection Criteria												
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Std. Dev. for in run Duplicates												

Precision												
-----------	--	--	--	--	--	--	--	--	--	--	--	--

Units					mg/L	mg/L						
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STRONTIUM

Strontium occurs in nature as the minerals celestite and strontianite. It is not found as a native metal but it does exist in the environment as a sulphate or in combination with calcium and barium minerals. Natural compounds of strontium are relatively insoluble; therefore, its presence in domestic water supplies is generally very low. Strontium-88 is derived from weathering of strontium minerals and sedimentary rocks bearing sulphate and carbonate. The testing of nuclear bombs has caused fallout of the radioactive Strontium-90 isotope. Industrial applications of strontium and its compounds include the manufacture of metal alloys, storage batteries, paints, rubber, cathode ray tubes, tracer bullets and flares; its use, however, is not widespread. The radioactive isotope Sr-90 is one of the best Beta-emitters known, and thus provides a light-weight, long lived nuclear energy source.

The chemical behavior of strontium closely resembles that of calcium with a tendency to become concentrated in the human skeletal system. There are no known harmful effects from oral ingestion. It does not appear to be necessary for plant nutrition. There are no water quality guidelines set for non-radioactive strontium.

Sampling

Special acid washed plastic containers are required for trace level determinations in domestic water and surface water samples. Glass containers are required for domestic waste and industrial waste samples. Add 20 drops of HNO_3 per liter. Soil samples should be collected in pomade jars. Vegetation samples should be collected in plastic bags and refrigerated. Fish samples should be placed in plastic bags and frozen.

Method

Samples are digested using an appropriate acid mixture and analyzed by atomic absorption.

Time of Analysis

Approximately 50 strontium analyses can be performed per day.

STRONTIUM	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	400 mL	400 mL		50 mL	100 mL	10 mL	2 grams	2 grams	50 grams			
Preferred Sampling Container	Plastic bottle Acid washed	Plastic bottle Acid washed		Glass bottle	Glass bottle	Glass bottle	Pomade jar	Plastic bag	Plastic bag			
Preservation Technique	HNO ₃ pH <2	HNO ₃ pH <2		HNO ₃ pH <2	HNO ₃ pH <2	None	None	4°C	Freeze			
Operating Range	0.001 to 0.25	0.001 to 0.25		0.02 to 5.0	0.01 to 2.5	0.10 to 25	*	*	*			
Limit Detection Criteria	0.001	0.001		0.02	0.01	0.10						
Std. Dev. for in run Duplicates												
Precision	0.015 at 0.20	0.015 at 0.20		0.20 at 2.0	0.015 at 0.20	-						
Units	mg/L	mg/L		mg/L	mg/L	mg/L	µg/g	µg/g	µg/g			

* = Test available upon request.

SULPHATE

Sulphate is an ionic component of all natural waters and is widely distributed in nature. It normally varies between 10 and 80 mg/L in surface waters, but may reach several thousand mg/L where industrial discharges are involved. These discharges include mine drainage wastes, tanneries, sulphite-pulp mills, textile plants, sulphuric acid plants and some metal working industries. Precipitation may contain considerable amounts of sulphate if industrial processes, such as the combustion of coal, emit large amounts of sulphur dioxide and sulphur trioxide to the atmosphere.

Waters high in sulphate tend to form hard scales in boilers and plumbing. Under anoxic conditions, sulphates serve as an oxygen source for bacteria which convert it to hydrogen sulphide gas. Sulphate is also believed to be responsible for "crown" corrosion in concrete sewer pipes.

The maximum sulphate concentration permissible for domestic supplies in Ontario is 250 mg/L. Concentrations above this level are non-toxic but sodium and magnesium sulphate exert a cathartic effect on the gastrointestinal tract. Waters above 500 mg/L may impart a bitter taste to the water as well as causing cathartic effects. Sulphate levels are generally not high enough to harm aquatic life.

Sampling

Surface, sewage, industrial waste and drinking water samples should be collected in glass bottles, while precipitation samples should be collected in polystyrene bottles. Soil and sediment samples should be collected in pomade jars.

Method

Analyses on surface, ground-water and drinking water, sewage, industrial wastes, Hi-Vol, dustfall and solid samples after extraction are performed colorimetrically on an AutoAnalyzer system using barium chloride and methyl thymol blue. Interferents are phosphate, sulphide, sulphite, humates, lignins and tannins. Precipitation, Lo-Vol sequential filters and low-level surface samples are analyzed by ion chromatography.

Time of Analysis

Approximately 200 sulphate tests can be performed per day by the colorimetric method and 20 by ion chromatography.

SULPHATE	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Vegh	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	50 mL	50 mL	50 mL	50 mL	50 mL		50 grams			5 mL	1 filter strip	
Preferred Sampling Container	Glass or polystyrene	Glass bottle	Polystyrene bottle	Glass bottle	Glass bottle		Pomade jar			Sampling jar		
Preservation Technique	None	None	4°C	None	None		None			None	None	
Operating Range	1.0 to 50	0.7 to 100	0.06 to 10	0.7 to 100	0.7 to 100		7 to 1000			625 to 100,000	0.1 to 18	
Limit Detection Criteria	1.77	1.38	0.112	1.38	1.38		13.8				0.1	
Std. Dev. for in run Duplicates	0.539 0.536 0.673	0.42 0.59 0.57	0.034 0.078 0.167	0.4 0.59 0.57	0.59 0.57		6.9					
Precision											0.3 at 15	
Units	mg/L SO ₄	mg/L SO ₄	mg/L SO ₄	mg/L SO ₄	mg/L SO ₄		µg/g			µg/m ² /30 days	µg/m ³	

SULPHATION RATE

This method is used for the determination of the sulphation rate of the atmosphere. The sulphation rate is a measure of oxidizable sulphur's reactivity, and therefore allows evaluation of the potential damaging ability of sulphur compounds. The sulphur compounds, particularly, sulphur dioxide, are converted to sulphate by a lead dioxide coated glass fibre filter. Climatic differences, interferences, differences in reactivity factors for lead dioxide batches and plate preparation techniques contribute to variable reactivities for different plates. Thus, it is difficult to compare one station to another.

Sampling

Samples are collected on a plate (lead dioxide coated glass fibre filter). These filters should be kept in a sealed container before and after use.

Method

The sulphate is separated from the other materials and analyzed colorimetrically using the methyl thymol blue technique. Results are reported as $\text{mg SO}_3/100 \text{ cm}^2/\text{day}$.

Sulphuric acid mist, particulate sulphate and sulphur trioxide may impinge upon the plate giving high results. Mercaptans act as negative interferences. All interferences with the methyl thymol blue test outlined in the sulphate section apply here.

Time of Analysis

A plate requires approximately 30 days of exposure and a week for analysis.

NOTE: This test is now done only at the Thunder Bay laboratory.

Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Vegh	Bio Material	Dust Fall	Hi-Vol	Ambient Air
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30 days exposure

Keep sealed

in use
0.03

2.14
0.05

$$\frac{\text{mg}/100 \text{ cm}^2}{\text{day}}$$

1

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Method

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Sulphuric acid mist, particulate sulphate and sulphur trioxide may impinge upon the plate giving high results. Mercaptans act as negative interferences. All interferences with the methyl thymol blue test outlined in the sulphate section apply here.

Time of Analysis

A plate requires approximately 30 days of exposure and a week for analysis.

NOTE: This test is now done only at the Thunder Bay laboratory.

SULPHATION RATE	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size												30 days exposure
Preferred Sampling Container												Plate
Preservation Technique												Keep sealed when not in use
Operating Range												0.03 to 2.14
Limit Detection Criteria												0.05
Std. Dev. for in run Duplicates												
Precision												
Units												$\frac{\text{mg}}{100 \text{ cm}^2 \text{ day}}$ SO ₂

SULPHIDE

Sulphide is formed by bacterial reduction of sulphate and organic sulphur compounds under anaerobic conditions. It is, therefore, commonly found in domestic wastewater, industrial wastewater, sludges, hypolimnions of stratified lakes and any other aquatic systems where anaerobic conditions prevail. As a result, concentrations in surface waters are negligible. Hydrogen sulphide may be found in various forms depending on the pH of the water. At pH 5, it is almost entirely found as H_2S while at pH 9, it is almost entirely HS^- . The S^{2-} anion is generally not found in natural waters. Its characteristic odour of "rotten eggs" is highly objectionable.

Sulphide is an important parameter in waste treatment monitoring. Oxidation of sulphide to sulphuric acid in concrete sewer pipes leads to "crown corrosion". Soluble sulphides in excess of 200 mg/L are toxic to bacteria and will inhibit sludge digestion. Concentrations of undissociated hydrogen sulphide should not exceed 0.002 mg/L at any time or place to protect aquatic life.

Samples containing sulphides are unstable and results must be interpreted with caution.

Sampling

Samples must be collected with a minimum amount of agitation as sulphides are highly volatile and susceptible to oxidation by dissolved oxygen. The container should be filled completely so that no air bubbles remain after the sample is capped. Immediate delivery to the laboratory is essential. Where possible a field analysis should be performed.

Preservation is accomplished by adding 2 mL of a 2N zinc acetate solution to the sample followed by the dropwise addition of a 5% sodium carbonate solution until precipitation is complete. Consultation with laboratory staff is recommended when sulphide analyses are required. Soil samples should be collected in pomade jars.

Method

Samples are acidified and analysis is accomplished colorimetrically usually after inert gas displacement of H_2S using the ammonium molybdate or methylene blue methods. The semi-quantitative lead acetate method is only used to obtain a rough estimate of the sulphide concentration. The ammonium molybdate-gas displacement method is preferred for most sample types because of its simplicity. Caution must be exercised in interpreting the data as some organic sulphur compounds may release sulphide under the test conditions. Sulphide results are reported in mg/L as H_2S .

Samples may be analyzed in the field using a Hach kit. Methylene blue without gas displacement is practicable for large numbers of relatively clean samples.

Time of Analysis

Approximately 15 sulphide analyses may be completed per day using the ammonium molybdate method.

SULPHIDE	Ground & Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	1000 mL	25 mL		1000 mL	1000 mL	50 grams	2 grams					
Preferred Sampling Container	Glass bottle	Glass bottle		Glass bottle	Glass bottle	Glass bottle	Pomade jar					
Preservation Technique	See text 4°C	See text 4°C		See text 4°C	See text 4°C	See text 4°C	None					
Operating Range	1 to 15	<5		1 to 15	1 to 15	*	*					
Limit Detection Criteria	0.02	0.1		0.02	0.02							
Std. Dev. for in run Duplicates												
Precision	0.2 at 2			0.2 at 2	0.2 at 2							
Units	mg/L	mg/L		mg/L	mg/L	mg/L	µg/g					

* = Test available upon request.

SUSPENDED AIR PARTICULATE

Suspended particulate matter in the atmosphere is present naturally and as a result of anthropogenic activity. Natural sources include wind blown dust, volcanic ash, pollens and smoke and fly ash from forest fires. Man's activities have greatly increased levels of suspended particulates, with the highest concentrations found in industrial areas, expressways, downtown areas and in the vicinity of solid waste incinerators. Residential and rural locations report relatively low levels of air particulate matter. Siliceous material generally comprises the largest component of total suspended particulate; however, the nature of the material depends primarily upon location and it is therefore difficult to generalize.

Suspended particulate in the atmosphere can cause a reduction in visibility, alterations in sky illumination due to changes in the light scattering pattern, a decrease in the amount of solar radiation reaching the earth's surface, damage to vegetation and property and physiological reactions including allergies and irritations to the respiratory tract. Health agencies have determined that particles of less than 5 μm in diameter can enter the respiratory tract and cause health problems. The accepted standard for total suspended particulate matter in the atmosphere in Ontario is 120 $\mu\text{g}/\text{m}^3$ for 24 hours, with a geometric mean of 60 $\mu\text{g}/\text{m}^3$ for 1 year.

Sampling

Samples are collected on tared glass fibre filter paper allowing the collection of particulate having an aerodynamic diameter of less than 100 μm . Samples are collected by means of a high-flow rate blower drawing air in at a rate of 40 - 60 cfm. Exposed filters are carefully removed from the holder, folded across the middle of the 10 inch side, placed in suitably marked envelopes and mailed to the laboratory. The sample should be kept dry at all times.

Method

The high volume glass fibre filter method has been accepted by most North American agencies as the standard medium for the determination of total suspended particulate, although other filters such as Whatman No. 41 and Microsorban may be used for special analyses.

Time of Analysis

Approximately 56 filters can be prepared, conditioned and analyzed per day.

[illegible]

TANNINS AND LIGNINS

Tannins and lignins are members of a large group of hydroxylated aromatic compounds. They are natural components of plant material and are found in surface waters as a result of normal biochemical degradation processes, or as a result of industrial discharges. They are commonly found in the effluents of the paper and tanning industries. Tannins are also applied internally to boilers in order to facilitate the handling of calcium sludge deposits and to prevent the formation of a hard scale. Tannins and lignins are undesirable because they impart objectionable tastes and colors to domestic water supplies even at very low levels.

At present no separations or individual determinations of tannins and lignins are performed. Results are standardized and reported as mg/L of tannic acid. To convert this result to a lignin number, the tannic acid value should be multiplied by 2.5.

Sampling

Since tannins and lignins are subject to normal biochemical oxidation, samples should be refrigerated to retard bacterial activity. No chemical preservative should be used as this may interfere with the analysis. Immediate delivery to the laboratory is essential.

Method

Tannins and lignins in aqueous samples are determined colorimetrically by direct treatment with tungstophosphoric and molybdophosphoric acids (tyrosine reagent). As other reducing agents produce the same color, their presence may cause an over-estimation of the actual tannin and lignin content. Solid samples undergo a leaching step before colorimetric analysis.

Time of Analysis

Approximately 40 tannin and lignin analyses may be completed in one day.

TANNINS AND LIGNINS	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	200 mL				200 mL							
Preferred Sampling Container	Glass bottle				Glass bottle							
Preservation Technique	4°C				4°C							
Operating Range	1.0 to 10				1.0 to 10							
Limit Detection Criteria	1.0				1.0							
Std. Dev. for 20-50% in run Duplicates												
Precision	1.0				1.0							
Units	mg/L				mg/L							

THALLIUM

Thallium occurs in the minerals crooksite, lorandite and hutchinsonite. It is also present in pyrites. Generally, the only commercial sources of thallium are flue dusts and residues from metallurgical and chemical processes such as smelting of lead and zinc ores in which thallium is a minor impurity. Thallium is generally present in only trace amounts in surface waters. The monovalent ion is the most stable form in aqueous solution.

Thallium readily alloys with a number of metals to form a number of low melting binary and higher order alloys. It has some applications in the electronics and glass industries. Thallium sulphate was an important ingredient in rodenticides and pesticides, but its toxicity to humans has curtailed this use.

The element and its compounds are toxic and should be handled carefully. Contact of the metal with skin is dangerous and may be carcinogenic to humans. Thallium has been used to treat ringworm and other skin infections but its use is limited because the margin of safety between therapeutic and toxic doses is too narrow. Thallium can inhibit photosynthetic processes and plant transpiration. In both fish and invertebrates it acts as a neuro-poison. The 96 hour LC 50 for rainbow trout is less than 10 mg/L. No drinking or livestock water guidelines have been set.

Sampling

Special acid washed plastic containers are required for trace level determinations in domestic water and surface water samples. Glass containers are required for domestic waste and industrial waste samples. Add 20 drops of HNO_3 per liter. Soil samples should be collected in pomade jars. Vegetation samples should be collected in plastic bags and refrigerated. Fish samples should be placed in plastic bags and frozen.

Method

The sample is digested with an appropriate acid mixture and analyzed by atomic absorption or polarograph.

Time of Analysis

Approximately 50 thallium samples can be analyzed per day.

THALLIUM	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	400 mL	400 mL	25 mL		400 mL	25 mL	2 grams	2 grams	50 grams			
Preferred Sampling Container	Plastic bottle Acid washed	Plastic bottle Acid washed	Plastic bottle Acid washed		Glass bottle	Glass bottle	Pomade jar	Plastic bag	Plastic bag			
Preservation	HNO ₃ pH <2	HNO ₃ pH <2	HNO ₃ pH <2		HNO ₃ pH <2	None	None	4°C	Freeze			
Technique												
Operating Range	0.01 to 0.10	0.01 to 0.10	*		0.01 to 0.10	*	*	*	*			
Limit Detection Criteria												
Std. Dev. for 20-50% in run Duplicates												
Precision	0.06 at 0.50	0.06 at 0.50			0.06 at 0.50							
Units	mg/L	mg/L	mg/L		mg/L	mg/L	µg/g	µg/g	µg/g			

* = Test available upon request.

TIN

Tin is found mainly in the mineral cassiterite. Most of the world's supply comes from Malaysia, Bolivia, Indonesia, Nigeria and Thailand. Tin is derived naturally from weathering processes. Tin is readily adsorbed by clay minerals so it is generally only found in trace amounts. The major sources of tin in the environment are industrial discharges.

Tin foil is used for electrical condensers, bottle cap liners and food wrappings. Tin wire is used for fuses and safety plugs and is used in the production of plate glass. Tin plating over steel is the most important use of tin. Tin cans in the food packaging are the major products. Tin alloys may be used in superconductive magnets. Tin also has a number of organic applications.

Tin and its inorganic compounds are essentially non-toxic. The amounts tolerated by the human body are far higher than would normally be ingested by eating canned foods. Many organic tin compounds are toxic, and may in addition be irritating to skin, mucous membrane and other tissues. Diethyl tin can cause severe lesions and even death. There are no guidelines for inorganic tin in drinking water. Since tin is not generally taken up by plants there are no agricultural guidelines.

Sampling

Special acid washed plastic containers are required for trace level determinations in domestic water samples. Glass containers are required for domestic waste and industrial waste samples. Add 20 drops of HNO_3 per liter.

Method

The sample is digested with an appropriate acid mixture and analyzed by flameless atomic absorption.

Time of Analysis

Approximately 50 tin analyses can be performed per day.

TIN Surface Water Drinking Water Precip. Sewage Ind. Wastes Sludge Soil & Sediment Veg'n Bio Material Dust Fall Hi-Vol Ambient Air

Minimum Sample Size

400 mL 400 mL 50 mL 100 mL 10 mL

Preferred Sampling Container

Plastic bottle Acid washed Plastic bottle Acid washed Glass bottle Glass bottle Glass bottle

Preservation Technique

HNO₃ pH <2 HNO₃ pH <2 HNO₃ pH <2 HNO₃ pH <2 None

Operating Range

0.04 to 5.0 0.04 to 5.0 0.8 to 5.0 0.4 to 25 4.0 to 200

Limit Detection Criteria

0.04 0.04 0.8 0.4 4.0

Std. Dev. for in run Duplicates

<20%
20-50%
50-100%

Precision

0.1 at 1.0 0.1 at 1.0 - 4.0 at 10 -

Units

mg/L mg/L mg/L mg/L mg/L

TITANIUM

Titanium is found in nature as the minerals rutile, ilmenite, and sphene. It ranks eighth in the natural abundance of elements and is readily found in most soils, varying between 0.5% and 10%. The high tensile strength and corrosion resistant properties of titanium metal are valuable in the manufacture of special steel alloys which are used for structural purposes in aircraft frames and engines. Titanium metal is also used in the manufacture of electrodes, lamp filaments, and X-ray tubes. Oxides of titanium are used as a white pigment in the paint, glass, and ceramics industries. Titanium and its salts are generally extremely inert and insoluble; the notable exception is titanium tetrachloride, which is highly corrosive.

Neither plants nor animals accumulate titanium to any extent. There is no evidence that titanium plays an essential role in human or animal nutrition.

There is no known toxicity to humans, animals, or plants. Surgical appliances are generally made of titanium and various titanium derivatives have been used in the treatment of skin disorders. No limits have been specified for the maximum acceptable concentration of titanium in domestic water supplies.

Sampling

Special acid washed plastic containers are required for trace level determinations in domestic water and surface water samples. Glass containers are required for domestic waste and industrial waste samples. Add 20 drops of HNO_3 per liter. Pomade jars should be used for soil and sediment samples. Vegetation samples should be collected in plastic bags and refrigerated. Fish samples should be placed in plastic bags and frozen.

Method

Samples are digested with an appropriate acid mixture and analyzed by atomic absorption.

Time of Analysis

Approximately 50 titanium analyses can be performed per day.

TITANIUM	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	400 mL	400 mL	25 mL	50 mL	100 mL	10 mL	2 grams	2 grams	50 grams			
Preferred Sampling Container	Plastic bottle Acid washed	Plastic bottle Acid washed	Plastic bottle Acid washed	Glass bottle	Glass bottle	Glass bottle	Pomade Jar	Plastic bag	Plastic bag			
Preservation Technique	HNO ₃ pH <2	HNO ₃ pH <2	HNO ₃ pH <2	HNO ₃ pH <2	HNO ₃ pH <2	None	None	4°C	Freeze			
Operating Range	0.04 to 0.50	0.04 to 0.50	*	0.8 to 10	0.4 to 5.0	4.0 to 50.0	10 to 2500	10 to 2500	*			
Limit Detection Criteria	0.04	0.04		0.8	0.4	4.0	10	10				
Std. Dev. for 20-50% in run Duplicates												
Precision	0.025 at 0.2	0.025 at 0.2		0.5 at 4	0.5 at 4	-	90 at 420	90 at 420				
Units	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	μg/g	μg/g	μg/g			

* = Test available upon request.

TRACER DYES

Sodium fluorescein is used as a tracer dye to follow ground water movement. It may also be used to monitor effluent from septic tank systems, especially where there is a suspected contamination on drinking water. Concentrations can be reported but the test is generally only used to detect the presence of the dye. Rhodamine dye can also be determined on special request.

Sampling

Samples should be collected in glass bottles and kept in a cool, dark place to minimize photosynthetic activity and oxidation.

Method

Analysis is performed using a fluorospectro-photometric technique.

[illegible]

TRIAZINE HERBICIDES

Triazine herbicides are used mainly on corn fields. This test presently detects the following substances: atrazine, simazine, propazine, bladex, prometryne, ametryene, prometryene, sencor and atratone.

Widespread use of these herbicides has led to considerable research into its toxicity and attenuation in soil. There is indirect evidence that s-triazines are rendered non-toxic in soils by microorganisms. Plants can metabolise the s-triazines but the rates of degradation vary from plant to plant.

From a practical standpoint, any regulations imposed on s-triazines should depend on soil and climatic conditions, crop sensitivity, the rate of application and the type of compound used. Detoxification may take place more quickly in the presence of crops than in their absence.

The maximum permissible levels are 100 $\mu\text{g/L}$ in the raw water supply, however recommended levels for raw waters are 7.5 $\mu\text{g/L}$ for Atrazine, 75 $\mu\text{g/L}$ for Simazine and 16 $\mu\text{g/L}$ for Propazine. Simazine levels should not exceed 10 $\mu\text{g/L}$ in an unfiltered sample to protect aquatic life. Levels for other compounds in this group are not specified. Irrigation waters should not have residues of triazines in excess of 0.05 $\mu\text{g/L}$ to avoid crop damage.

Bladex has been designated as a high ecological hazard, promatone and simazine as moderate hazards and atrazine as a slight hazard by the Environmental Protection Service.

Sampling

Aqueous samples should be collected in solvent rinsed brown glass bottles with foil lined caps (Pack 3P). Bottles should be filled to the mark. Solid samples should be collected in solvent rinsed jars (Pack 5P). All samples should be refrigerated and transported to the laboratory as quickly as possible.

Method

Samples undergo solvent extraction, evaporation, clean-up with alumina and analysis by N/P gas chromatography.

Time of Analysis

Approximately 8 analyses can be performed per day.

TRIAZINE
HERBICIDES

Minimum Sample Size 800 mL 800 mL 800 mL 10 grams 10 grams

Preferred Sampling Container Special bottle Special bottle Special bottle Special jar Special jar

Preservation Technique 4°C 4°C 4°C 4°C 4°C

Operating ** Range >0.05 >0.05 >0.05 >0.05 >0.05

Limit Detection Criteria 0.05 to 0.10 0.05 to 0.10 0.05 to 0.10 0.05 to 0.10 0.05 to 0.10

Std. Dev. for in run Duplicates < 20% 20-50% 50-100%

Precision * * * * *

Units µg/L µg/L µg/L µg/g µg/g

* = Replicate analyses should be within 10% of the mean.
** = Actual operating range may vary from the detection limits shown to percent levels.

TURBIDITY

Materials present in water absorb, scatter and reflect light as it passes through it. Dissolved materials absorb light but a substantial reduction in light transmission is due to the presence of suspended particles. Turbidity is a measure of the optical properties of a sample which cause light to be scattered rather than transmitted in straight lines through the sample. In water samples, turbidity is a result of suspended clay, silt, finely divided organic and inorganic matter, plankton and other microscopic organisms. While shoreline erosion, resuspension of sediment material and algal blooms decrease light transmission naturally, human activities can also have an effect.

Turbidity cannot be successfully correlated with the suspended solids test because the size, shape, refractive index and hence, the light scattering effect of the suspended matter have little relationship to their concentration and specific gravity.

Water clarity is one of the main criteria used by the public to judge water quality for drinking and recreational use.

Care must be used in interpreting turbidity results since these are defined by the instrument, the standards and the optical phenomena being measured. Standards other than Formazin are exceedingly difficult to reproduce as their optical properties depend on the sizes, shapes and refractive indices of the component particles in suspension.

The turbidity of the water can have a great effect on the types and quantities of algae that grow in it by altering the amount of light available for photosynthesis. Suspended matter should not be added to surface waters in concentrations that will change the natural secchi disc reading by more than 10 percent.

Sampling

Samples should be collected in glass or polystyrene bottles. Samples should be kept in the dark and delivered to the laboratory within 24 hours.

Method

Light scattered by the sample at $90^\circ \pm 30^\circ$ is measured in a nephelometer calibrated with Formazin standards. Results are reported in Formazin Turbidity Units (FTU) or Nephelometric Units (NTU). Rapid settling particles or gross debris may cause erroneous results. Color due to dissolved substances which absorb light will cause low results.

Time of Analysis

Approximately 150 turbidity determinations can be performed per day.

TURBIDITY	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	50 mL	50 mL		50 mL	50 mL							
Preferred Sampling Container	Glass or poly-styrene	Glass or poly-styrene		Glass bottle	Glass bottle							
Preservation Technique	Keep in dark	Keep in dark		Keep in dark	Keep in dark							
Operating Range	0.2 to 10	0 to 10		0 to 10	0 to 10							
Limit Detection	0.418	-		-	-							
Detection Criteria	0.209											
Std. Dev. for in run Duplicates	< 20% 0.127 20-50% 0.247 50-100% 0.278	- - -		- - -	- - -							
Precision												
Units	FTU	FTU		FTU	FTU							

FTU = Formazin Turbidity Units

URANIUM

Uranium is found in nature as the minerals carnotite and uraninite as well as in igneous type rocks. It is well known for its radioactive properties since its major industrial application is the manufacture of nuclear fuel. Natural uranium, however, is present as the isotope U-238, which is not strongly radioactive. Uranium in natural waters is derived from weathering of uranium minerals and acid igneous rocks and from discharges from nuclear fuel processing plants and from photography and glazing industries.

In natural waters uranium is usually hexavalent and commonly present as the uranyl ion UO_2^{++} . Uranyl salts are very soluble and produce an objectionable taste and color when they exceed a concentration of 5 mg/L as uranium. At considerably higher levels, ingestion of water containing uranyl ions may result in possible kidney damage. Limits on the maximum concentration of uranium in domestic water supplies, when specified, are generally based on chemical properties of the uranyl ion rather than on radioactivity.

Sampling

Special acid washed plastic containers are required for trace level determinations in domestic water and surface samples. Glass containers are required for domestic waste and industrial waste samples. Add 20 drops of HNO_3 per liter.

Method

Samples are analyzed by ICP atomic emission.

Time of Analysis

Approximately 50 uranium analyses can be performed per day.

URANIUM	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	400 mL	400 mL			400 mL							
Preferred Sampling Container	Plastic bottle Acid washed	Plastic bottle Acid washed			Glass bottle							
Preservation Technique	HNO ₃ pH <2	HNO ₃ pH <2			HNO ₃ pH <2							
Operating Range	0.05 to 1000	0.05 to 1000			0.05 to 1000							
Limit Detection Criteria	0.05	0.05			0.05							
Std. Dev. for in run Duplicates												
Precision	*	*			*							
Units	mg/L	mg/L			mg/L							

* = ± 10% at 10x Detection Limit.

VANADIUM

Vanadium occurs in nature as the minerals vanadinite, patronite, mottramite, and carnotite, and is generally found in the presence of selenium. Metallic vanadium does not occur in nature. Trivalent vanadium salts tend to be insoluble but tetra- and pentavalent salts tend to be soluble. Seepage from carbonaceous deposits may be a major source of vanadium. Other sources include weathering and industrial discharges. Vanadium is used in the steel industry where it is known to increase the general toughness and tensile strength of certain alloys, but it is also used in the manufacture of dyes, paints, inks and ceramics. Vanadium has been found in all fuel and motor oils and has been used for oil spill identification by means of the nickel-vanadium ratio. Vanadium in surface waters is generally found at levels below 0.05 mg/L.

The usefulness of vanadium in animal and plant metabolism has not been convincingly demonstrated and its behaviour in biological systems is not well understood. Some experimental evidence suggests that the presence of vanadium in trace quantities may be beneficial in the prevention of heart disease.

Several compounds of vanadium are known to be toxic in high concentration. Exposure may produce nausea, diarrhea, abdominal pain, nervous trembling of the hands, and greenish-black discoloration of the tongue. No limits have been specified for the maximum concentration of vanadium in domestic water supplies because the element is rarely encountered. Levels in livestock drinking waters should not exceed 0.1 mg/L.

Sampling

Special acid washed plastic containers are required for trace level determinations in domestic water and surface water samples. Glass containers are required for domestic waste and industrial waste samples. Add 20 drops of HNO_3 per liter. Pomade jars should be used for soil and sediment samples. Vegetation samples should be collected in plastic bags and refrigerated. Fish samples should be placed in plastic bags and frozen.

Method

Samples are digested using the appropriate acid mixture and analyzed. Hi-Vol samples are analyzed by ICP atomic emission. All other samples are analyzed by atomic absorption.

Time of Analysis

Approximately 50 vanadium analyses may be performed per day.

VANADIUM	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	400 mL	400 mL	25 mL	50 mL	100 mL	10 mL	2 grams	2 grams	50 grams	200 mL	1 filter strip	
Preferred Sampling Container	Plastic bottle Acid washed	Plastic bottle Acid washed	Plastic bottle Acid washed	Glass bottle	Glass bottle	Glass bottle	Pomade jar	Plastic bag	Plastic bag	Sampling jar	Envelope	
Preservation Technique	HNO ₃ pH <2	HNO ₃ pH <2	HNO ₃ pH <2	HNO ₃ pH <2	HNO ₃ pH <2	None	None	4 °C	Freeze	None	None	
Operating Range	0.002 to 0.25	0.002 to 0.25	0.002 to 0.200	0.05 to 70	0.02 to 2.5	0.25 to 350	3 to 2500	5 to 100	*	0.12 to 2.0	0.0002 to 0.35	
Limit Detection Criteria	0.002	0.002	0.002	0.05	0.02	0.25	3	5		0.12	0.0002	
Std. Dev. for in run Duplicates												
Precision	0.017 at 0.20 mg/L	0.017 at 0.20 mg/L	0.0003 at 0.004 mg/L	0.425 at 5 mg/L	0.017 at 0.20 mg/L	2.1 at 25 mg/L	2.5 at 58.6 µg/g	4 at 50 µg/g		-	**	
Units							µg/g	µg/g	µg/g	µg/m ² /30 days	µg/m ³	

* = Test available upon request
 ** = ± 10% at 10x Detection Limit.

VINYL CHLORIDE (VCM)

Vinyl chloride monomer ($\text{CH}_2 = \text{CHCl}$) is a colorless, odourless gas which is slightly soluble in water at normal temperatures. It is the base material for polyvinyl chloride (PVC) thermoplastic resins which can be fabricated into products such as electrical insulation, wrapping foils, plumbing tubing and pipes. It has some use as a solvent extractant for heat soluble substances, as a refrigerant, and as an aerosol propellant.

VCM is only produced by industrial processes. It is not a naturally occurring compound. Ministry guidelines allow maximum VCM levels in ambient air of 0.1 mg/L (v/v) averaged over 25 hours and 0.2 mg/L (v/v) averaged over 0.5 hours at a point of impingement relative to an industrial source. It is a suspected carcinogen.

Sampling

Aqueous samples should be collected in 1 liter glass bottles with foil lined caps. Solid samples should be collected in pomade jars. Air samples may be collected in polyester-protected aluminized bags or in charcoal adsorption tubes. Aqueous, solid and tube samples should be kept cool and transported to the laboratory as quickly as possible. Sampling bags should be kept at room temperature and protected from sunlight.

Method

Bag samples are analyzed by gas chromatography after direct injection. Adsorption tube samples are subjected to desorption with carbon disulphide prior to injection into the gas chromatograph. Solid samples are analyzed by gas chromatography after head space analysis. Aqueous samples are analyzed by direct injection if feasible or by head space analysis of the vapours in sealed bottles fitted with "Teflon" septa.

VINYL CHLORIDE

Minimum Sample Size	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
	1000 mL	1000 mL	1000 mL	1000 mL	1000 mL	20 grams	20 grams	20 grams	20 grams			48 hour ex- posure for tube or half full bag
Preferred Sampling Container	Glass bottle	Glass bottle	Glass bottle	Glass bottle	Glass bottle	Glass bottle	Pomade jar	Pomade jar	Pomade jar			Charcoal tube or sampling bag
Preservation Technique	4°C	4°C	4°C	4°C	4°C	4°C	4°C	4°C	4°C			Keep in dark
Operating Range	>10	>10	>10	>10	>10	>1.0	>1.0	>1.0	>1.0			>100
Limit Detection Criteria	10	10	10	10	10	1.0	1.0	1.0	1.0			100
Std. Dev. for in run Duplicates												
Precision	*	*	*	*	*	*	*	*	*			*
Units	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/g	µg/g	µg/g			µg/m³

* = Replicate analyses should be within 10% of the mean.

VOLATILE ACIDS

Low molecular weight organic acids which are readily distilled from aqueous solution at low temperatures are commonly called volatile acids. They include acetic, propanoic, and butanoic acids as well as small amounts of valeric, isovaleric, and caprioc acids. These volatile acids are formed as intermediates during the anaerobic degradation of carbohydrates, proteins and fats in sludge treatment processes. An excess accumulation of these acids can have a deleterious effect upon anaerobic digestion. In routine operation of an anaerobic waste treatment system, the volatile acid concentration is primarily used as the determining and controlling factor for maintaining a satisfactory balance between the saprophytic and methane producing bacteria which are largely responsible for the efficiency of anaerobic digestion.

Volatile acids are found in natural waters when anoxic conditions are prevalent, but they are extremely unstable and would not likely be found in domestic water supplies.

Results are reported as mg/L of acetic acid.

Sampling

Sludges should be collected in wide mouth glass jars and not filled more than half way. As volatile acids are extremely unstable they should be maintained at 4 °C and delivered to the laboratory immediately.

Method

Volatile acids are separated by means of a column chromatographic technique using a chloroform-butanol eluant. The eluate is then titrated with standard alcoholic sodium hydroxide to the phenolphthalein endpoint. This procedure is capable of eluting higher molecular weight organic acids as well as some synthetic detergents; however, the concentration of these substances is generally negligible in sludges.

Time of Analysis

Approximately 20 volatile acid determinations may be completed per day.

VOLATILE ACIDS	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size				25 mL		25 mL						
Preferred Sampling Container				Glass bottle		Glass bottle						
Preservation Technique				4°C		4°C						
Operating Range				13 to 5000		13 to 5000						
Limit Detection Criteria				25.1		25.1						
				12.55		12.55						
Std. Dev. for in run Duplicates				7.63 8.51 -		7.63 8.51 -						
Precision												
Units				mg/L Acetic Acid		mg/L Acetic Acid						

ZINC

Zinc is commonly found in nature as zinc sulphide in the mineral sphalerite and as zinc carbonate in the mineral smithsonite. It is widely used in the manufacture of metal alloys such as brass, batteries, dyestuffs, paints, ointments and rubber, as well as in electroplating. Zinc oxide is used in medical ointments and as a pigment. Zinc carbonate and sulphide are used as white pigments in paints and enamels. The sulphide is also used as a phosphor for television screens and fluorescent coatings. Zinc most commonly enters a water supply via the deterioration of galvanized iron, the dezincification of brass, direct industrial discharges, and drainage from mine wastes. It is usually found in concentrations less than 0.05 mg/L in most natural waters but acidic waters may hold up to 50 mg/L.

Zinc is essential to plant and animal life. It is a component of several metalloenzymes and proteins. Deficiencies result in growth retardation, skin lesions, bone weaknesses, poor healing of wounds, impaired reproductive processes and reduced learning capacity.

Plants growing in a zinc deficient soil are severely stunted. In animals, zinc is an important constituent of a number of enzymes, notably carbonic anhydrase which is vital for normal respiration. Zinc and its compounds are relatively non-toxic when taken orally. Even at high concentrations, nausea and diarrhea are the only adverse effects likely to be encountered. The limiting factors which determine the acceptable maximum concentration in a water supply are taste and appearance. Zinc, in excess of 5 mg/L, imparts a bitter astringent taste when the water is boiled and may give a milky appearance in alkaline waters. The maximum acceptable limit for domestic water supplies in Ontario is therefore 5 mg/L. Concentrations of zinc in an unfiltered sample should not exceed 0.030 mg/L to protect aquatic life. Zinc toxicity increases with increasing temperature and decreasing dissolved oxygen. The presence of copper and cadmium enhance the sublethal toxicity of zinc.

Sampling

Special acid washed plastic containers are required for trace level determinations in domestic water and surface water samples. Glass containers are required for domestic waste and industrial waste samples. Add 20 drops of HNO_3 per liter. Pomade jars should be used for soil and sediment samples. Vegetation samples should be collected in plastic bags and refrigerated.

Method

Samples are digested with the appropriate acid mixture and analyzed. Sewage, sludge and Hi-Vol samples are analyzed by ICP atomic emission. All other samples are analyzed by atomic absorption.

Time of Analysis

Approximately 50 zinc analyses may be performed per day.

ZINC	Surface Water	Drinking Water	Precip.	Sewage	Ind. Wastes	Sludge	Soil & Sediment	Veg'n	Bio Material	Dust Fall	Hi-Vol	Ambient Air
Minimum Sample Size	400 mL	400 mL	25 mL	50 mL	100 mL	10 mL	2 grams	2 grams	50 grams	200 mL	1 filter strip	
Preferred Sampling Container	Plastic bottle Acid washed	Plastic bottle Acid washed	Plastic bottle Acid washed	Glass bottle	Glass bottle	Glass bottle	Pomade jar	Plastic bag	Plastic bag	Sampling jar	Envelope	
Preservation Technique	HNO ₃ pH <2	HNO ₃ pH <2	HNO ₃ pH <2	HNO ₃ pH <2	HNO ₃ pH <2	None	None	4 °C	Freeze	None	None	
Operating Range	0.001 to 0.25	0.001 to 0.25	0.002 to 0.200	0.05 to 70	0.01 to 2.5	0.25 to 350	1 to 500	1 to 200	0.125 to 0.30	0.12 to 30	0.004 to 0.35	
Limit Detection Criteria	0.001	0.001	0.002	0.05	0.01	0.25	1	1	0.125	0.12	0.004	
Std. Dev. for 20-50% in run Duplicates	0.01 at 0.20	0.01 at 0.20	0.0006 at 0.029	*	0.026 at 0.40	*	2.3 at 241	8 at 100	-	-	*	
Precision	0.01 at 0.20	0.01 at 0.20	0.0006 at 0.029	mg/L	mg/L	mg/L	mg/L	mg/L	µg/g	µg/m ² /30 days	µg/m ³	
Units	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	µg/g	µg/g	µg/g	µg/m ² /30 days	µg/m ³	

* = ± 10% at 10x Detection Limit



SPECIALIZED CAPABILITIES

In addition to the tests outlined in this manual there is available a high degree of specialized expertise and equipment for conducting project work and special studies. Such specialized work often requires a great deal of planning and is time consuming. It cannot be considered part of the routine work and may require specific consultations and funding.

Some specialized capabilities are listed below.

- Microscopic identification of materials (particulates, fungi, pollen, etc.)
- Metal scanning in various matrices by powder spectrograph and ICP atomic emission.
- Electron microscope and electron probe M-210 analysis of particulate matter in water and air, of soil, sediment and special materials including bacterial cells. All metals can be scanned qualitatively and semi-quantitatively.
- Polarographic procedures, such as differential pulse anodic stripping voltametry (ultra trace concentrations of certain metals, speciation of ions and complexation.
- X-ray diffraction identification of crystalline compounds.
- Gas chromatography/mass spectrometer analysis of organic compounds..
- Ion chromatograph scanning for inorganic anions in water, precipitation and air samples. This is a quantitative scan.
- Oxygen uptake measurements on wastes and sediments using incubated sampling vessels and oxygen producing probes.
- Analysis of component parts from a sampling train (probe filters, impinger fractions) inserted in a stack. These are usually analyzed for routine metals and anions.
- Biomass determinations (direct count, ATP and Coulter counter).
- Metabolic activity (respirometer, continuous culture).
- Microbiological transformation of environmental contaminants (batch culture, continuous culture, respirometer studies).
- Detection of genotoxic activity in pure substances and environmental samples. Methods now available include the Ames and Rosencrantz tests.
- Detection and isolation of viruses from natural waters, sediments and sewage samples.
- Determination of plasticity, permeability and cation exchange capacity for soils and sediments; settleability of dredged spoil.
- Analysis of extractions and leachates of soil, sediment and solid industrial waste samples.

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APPENDIX

TABLE I

ANALYTICAL TEST CAPABILITIES

Chemical Parameter	Surface Waters	Domestic Waters	Precip.	Sewage	Industrial Waste	Sludges	Soil & Sediment	Vegetation	Bio Materials	Dust Fall	Hi-Vol Filters	Ambient Air
Acidity	W, L, K, TB	W, L, K, TB	W	W, L, K, TB	W, L, K, TB		ITC*					
Aldehydes, Ketones	OTC	OTC		OTC	OTC	OTC	OTC	OTC	OTC			
Aliphatic Alcohols	OTC	OTC		OTC	OTC							
Alkalinity	W, L, K, TB	W, L, K, TB	W	W, L, K, TB	W, L, K, TB		ITC*					
Aluminum	ITC, L, TB	ITC, L, TB	ITC	ITC, TB	ITC, L, TB	ITC	ITC, TB	ITC, TB	ITC		ITC	
Ammonia Nitrogen	See Nitrogen, Ammonia and Ammonium											
Anionic Detergents	W	W		W	W	W						
Antimony	ITC	ITC					ITC*	ITC				
Aromatic Hydrocarbons	OTC	OTC		OTC	OTC	OTC	OTC	OTC	OTC			OTC
Arsenic	ITC, TB	ITC, TB		ITC, TB	ITC, TB	ITC	ITC, TB	ITC, TB	ITC			
Asbestos	W	W									W	
Barium	ITC	ITC		ITC	ITC	ITC	ITC	ITC*	ITC*			

W = Water Quality Section ITC = Inorganic Trace Contaminants Section OTC = Organic Trace Contaminants Section
M = Microbiology Section P = Pesticides Section L = London Laboratory (Southwestern Region)
K = Kingston Laboratory (Southeastern Region) TB = Thunder Bay Laboratory (Northwestern Region) * By Special Request only

ANALYTICAL TEST CAPABILITIES

Chemical Parameter	Surface Waters	Domestic Waters	Precip.	Sewage	Industrial Waste	Sludges	Soil & Sediment	Vegetation	Bio Materials	Dust Fall	Hi-Vol Filters	Ambient Air
Beryllium	ITC	ITC		ITC*	ITC*	ITC*	ITC*	ITC*	ITC*			
BOD ₅	W, L, K, TB	W, L, K, TB		W, L, K, TB	W, L, K, TB							
Boron	ITC	ITC		ITC	ITC	ITC	ITC*	ITC				
Cadmium	ITC, L TB	ITC, L TB	ITC	ITC TB	ITC, L TB	ITC	ITC TB	ITC TB	ITC	ITC	ITC	
Calcium	W, L, K, TB	W, L, K TB	W, ITC	ITC, L, K TB	ITC, L, K TB	ITC	ITC TB	ITC TB		ITC TB		
Carbamate Insecticides & Herbicides	P	P			P							
Carbon Dioxide	W	W										
Carbon - Dissolved Inorganic	W	W		W	W	W						
Carbon - Low Level Dissolved Inorganic	W*											
Carbon - Dissolved Organic	W	W		W	W	W						

W = Water Quality Section ITC = Inorganic Trace Contaminants Section OTC = Organic Trace Contaminants Section
M = Microbiology Section P = Pesticides Section L = London Laboratory (Southwestern Region)
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ANALYTICAL TEST CAPABILITIES

Chemical Parameter	Surface Waters	Domestic Waters	Precip.	Sewage	Industrial Waste	Sludges	Soil & Sediment	Vegetation	Bio Materials	Dust Fall	Hi-Vol Filters	Ambient Air
COD	W, L, K TB	W, L, K, TB		W, L, K, TB	W, L, K, TB	ITC*	ITC* W*					
Chloride	W, L, K TB	W, L, K TB	W	W, L, K TB	W, L, K TB	ITC	ITC	ITC		ITC	ITC	
Chlorine - Residual	W	W		W								
Chlorophenol Insecticides	P	P			P							
Chlorophyll	W, TB											
Chromium Total	ITC, L TB	ITC, L TB	ITC	ITC, TB	ITC, L TB	ITC	ITC TB	ITC TB	ITC	ITC	ITC	
Chromium - Hexavalent	ITC	ITC			ITC							
Cobalt	ITC, L TB	ITC, L TB		ITC TB	ITC, L TB	ITC	ITC TB	ITC TB	ITC	ITC		
Colour - Apparent	W, L, K TB	W, L, K TB			W, L, K TB							
Conductivity	W, L, K TB	W, L, K TB	W	W, L, K TB	W, L, K TB	ITC*	ITC					

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Chemical Parameter	Surface Waters	Domestic Waters	Precip.	Sewage	Industrial Waste	Sludges	Soil & Sediment	Vegetation	Bio Materials	Dust Fall	Hi-Vol Filters	Ambient Air
Copper	ITC, L TB	ITC, L TB	ITC	ITC TB	ITC, L TB	ITC	ITC TB	ITC TB	ITC	ITC	ITC	
Cyanide	ITC	ITC		ITC	ITC	ITC	ITC					
Dustfall										ITC, L TB		
Fluoridation Rate												TB
Fluoride	W, L, K TB	W, L, K TB		W	W	ITC	ITC	ITC		ITC	ITC	
Glycols	OTC	OTC		OTC	OTC							
Gold	ITC	ITC		ITC	ITC	ITC*	ITC*	ITC*	ITC*			
Hardness	W, L, K TB	W, L, K TB		W, L, K TB	W, L, K TB							
Hydrocarbon Gases	OTC	OTC		OTC	OTC	OTC	OTC	OTC	OTC			OTC
Iron - Total	W, ITC L, K, TB	W, ITC L, K, TB	ITC	ITC L, K, TB	ITC L, K, TB	ITC	ITC TB	ITC TB	ITC	ITC	ITC	

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Chemical Parameter	Surface Waters	Domestic Waters	Precip.	Sewage	Industrial Waste	Sludges	Soil & Sediment	Vegetation	Bio Materials	Dust Fall	Hi-Vol Filters	Ambient Air
Lead	ITC, TB L	ITC, TB L	ITC	ITC, TB	ITC, TB L	ITC	ITC, TB	ITC, TB	ITC	ITC	ITC	
Lithium	ITC	ITC		ITC*	ITC	ITC*	ITC*	ITC*	ITC*	ITC*		
Magnesium	W, L, K TB, ITC	W, L, K TB, ITC	W, ITC	ITC L, K, TB	ITC L, K, TB	ITC	ITC	ITC	ITC	ITC	ITC	
Manganese	W, TB ITC	W, TB ITC	ITC	ITC, TB	ITC, TB	ITC	ITC, TB	ITC, TB	ITC	ITC	ITC	
Matrix Elements in Vegetation								ITC				
Mercury	ITC, TB	ITC, TB	*	ITC, TB	ITC, TB	ITC	ITC, TB	ITC, TB	ITC			
Microbiological Indicators of Fecal Pollution	M, L, K, TB	M, L, K, TB		M, L, K, TB	*	*	*					*
1) E. col												
2) Fecal coliforms												
3) Fecal streptococci												
4) Presence-Absence												
5) P. Aeruginosa												
6) Salmonella												
7) Total coliforms												

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ANALYTICAL TEST CAPABILITIES

Chemical Parameter	Surface Waters	Domestic Waters	Precip.	Sewage	Industrial Waste	Sludges	Soil & Sediment	Vegetation	Bio Materials	Dust Fall	Hi-Vol Filters	Ambient Air
Microbiological Indicators of Organic Enrichment 1) Fungi (Yeasts and Molds) 2) Heterotrophic Bacteria - 20°C 3) Heterotrophic Bacteria - 35°C	M, L, K, TB	M, L, K, TB		M, L, K, TB	*	*	*					*
Microbiological Indicators of Specific (Industrial/ Agricultural) Pollution 1) K. pneumoniae 2) Nitrogen cycle Bacteria 3) Phenol degrading Bacteria 4) Sulphate Reducing Bacteria 5) Sulphur Oxidizing Bacteria	M, L, K, TB	M, L, K, TB		M, L, K, TB	*	*	*					*
Microbiological Nuisance Organisms	M	M		M	*	*	*					*

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ANALYTICAL TEST CAPABILITIES

Chemical Parameter	Surface Waters	Domestic Waters	Precip.	Sewage	Industrial Waste	Sludges	Soil & Sediment	Vegetation	Bio Materials	Dust Fall	Hi-Vol Filters	Ambient Air
Moisture Content												
Molybdenum	ITC, L	ITC, L	ITC*	ITC	ITC, L	ITC	ITC	ITC*	ITC*			
Musks	Gasoline and Fuel Oil											
Nickel	ITC, TB L	ITC, TB L	ITC	ITC, TB	ITC, TB L	ITC	ITC, TB	ITC, TB	ITC	ITC	ITC	
Nitrogen - Ammonia and Ammonium	W, L, K TB	W, L, K TB	W	W, L, K TB	W, L, K TB	W, L, K	ITC				W	
Nitrogen - Nitrate	W, L, K TB	W, L, K TB	W	W, L, K TB	W, L, K TB	W, L, K	ITC				W	
Nitrogen - Nitrite	W, L, K TB	W, L, K TB		W, L, K TB	W, L, K TB	W, L, K	ITC					
Nitrogen - Total Kjeldahl	W, L, K TB	W, L, K TB		W, L, K TB	W, L, K TB	W, L, K ITC	ITC	ITC				
Nitrotoluenes	OTC	OTC		OTC	OTC							OTC
Organic Solvents	OTC	OTC		OTC	OTC	OTC	OTC	OTC	OTC			P
Organochlorine Pesticides and PCB's	P	P		P	P	P	P	P	P			

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ANALYTICAL TEST CAPABILITIES

Chemical Parameter	Surface Waters	Domestic Waters	Precip.	Sewage	Industrial Waste	Sludges	Soil & Sediment	Vegetation	Bio Materials	Dust Fall	Hi-Vol Filters	Ambient Air
Organohalides Volatile	OTC	OTC		OTC	OTC							
Organophosphorus Insecticides	P	P		P	P	P	P	P				
Organosulphur Compounds	OTC	OTC		OTC	OTC	OTC	OTC	OTC	OTC			OTC
Orthophosphate See: Phosphorus Filt. Reactive												
Oxygen - Dissolved	W, L, K TB											
Particle Size							ITC					
Petroleum Hydrocarbons	OTC	OTC		OTC	OTC	OTC	OTC					
pH	W, L, K TB	W, L, K TB	W	W, L, K TB	W, L, K TB	W TB	ITC TB			TB		
Phenolics	W, OTC L	W, OTC L		W, OTC L	W, OTC L		OTC					
Phenoxy Acid Herbicides	P	P		P	P							

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Chemical Parameter	Surface Waters	Domestic Waters	Precip.	Sewage	Industrial Waste	Sludges	Soil & Sediment	Vegetation	Bio Materials	Dust Fall	Hi-Vol Filters	Ambient Air
Phosphorus - Extractable	See Phosphorus in soils.											
Phosphorus Filtered Reactive	W, L, K TB	W, L, K TB		W, L, K TB	W, L, K TB							
Phosphorus Filtered Total	W											
Phosphorus in Soils Sediment & Vegetation							ITC	ITC				
Phosphorus Total	W, L, K TB	W, L, K TB		W, L, K TB	W, L, K TB	W, L, K TB	ITC	ITC	ITC*			
Phosphorus - Total By Dissolution	See Phosphorus in soils.											
Phthalic Acid Esters	OTC	OTC		OTC	OTC							
Polynuclear Aromatic Hydrocarbons	OTC	OTC		OTC	OTC	OTC	OTC	OTC	OTC	OTC	OTC	OTC
Potassium	W, L, K TB	W, L, K TB	W	W TB	W TB		ITC	ITC	ITC*			
Radionuclides	W	W										
Selenium	ITC	ITC		ITC	ITC	ITC	ITC	ITC	ITC			

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Chemical Parameter	Surface Waters	Domestic Waters	Precip.	Sewage	Industrial Waste	Sludges	Soil & Sediment	Vegetation	Bio Materials	Dust Fall	Hi-Vol Filters	Ambient Air
Silicon & Silicates - Reactive	W, TB	W, TB		W, TB	TB			ITC				
Silver	ITC	ITC		ITC	ITC	ITC	ITC*					
Sodium	W, L, K TB	W, L, K TB	W	W, L, K TB	W, L, K TB	ITC*	ITC	ITC		ITC TB		
Solids - Dissolved	W, L, K TB	W, L, K TB		W, L, K TB	W, L, K TB							
Solids - Ignited	W, L, K TB	W, L, K TB		W, L, K TB	W, L, K TB							
Solids - Suspended	W, L, K TB	W, L, K TB		W, L, K TB	W, L, K TB							
Solids - Total	W, L, K TB	W, L, K TB		W, L, K TB	W, L, K TB							
Strontium	ITC	ITC		ITC	ITC	ITC	ITC	ITC	ITC*			
Sulphate	W, L, K TB	W, L, K TB	W	W, L, K TB	W, L, K TB		ITC			ITC, TB	W	
Sulphation Rate												TB
Sulphide	ITC, L	ITC, L		ITC, L	ITC, L	ITC*	ITC*					

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Chemical Parameter	Surface Waters	Domestic Waters	Precip.	Sewage	Industrial Waste	Sludges	Soil & Sediment	Vegetation	Bio Materials	Dust Fall	Hi-Vol Filters	Ambient Air
Suspended Air Particulate											ITC TB	
Tannins & Lignins	OTC				OTC							
Thallium	ITC	ITC	ITC*		ITC	ITC*	ITC*	ITC*	ITC*			
Tin	ITC	ITC		ITC	ITC	ITC	ITC	ITC				
Titanium	ITC	ITC	ITC*	ITC	ITC	ITC	ITC	ITC	ITC*			
Tracer Dyes	OTC				OTC							
Triazine Herbicides	P	P			P	P	P	P				
Turbidity	W, L, K TB	W, L, K TB		W, L, K TB	W, L, K TB							
Uranium	ITC	ITC			ITC							
Vanadium	ITC	ITC	ITC	ITC	ITC	ITC	ITC	ITC	ITC*	ITC	ITC	
Vinyl Chloride	OTC	OTC		OTC	OTC	OTC	OTC	OTC	OTC			OTC
Volatile Acids				W		W						
Zinc	ITC, TB	ITC, TB	ITC	ITC, TB	ITC, TB	ITC	ITC, TB	ITC, TB	ITC	ITC	ITC	

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TABLE 2

MINIMUM AMOUNT OF SAMPLE REQUIRED

PARAMETER	Surface Water mL	Well Water mL	Precip. mL	Sewage mL	Ind. Waste mL	Sludge mL	Soil & Sediment grams	Veg'n grams	Bio Material grams	Dust Fall mL	Hi-Vol Air	Preferred Sampling Container	Preservation Technique
Acidity	100	50	100	50	50							Pyrex or Polystyrene Pomade jar	Refrigerate See Text
Aldehydes Ketones	1000	1000		1000	1000							Glass Bottle Pomade jar	Refrigerate Refrigerate
Aliphatic Alcohols	1000	1000		1000	1000	20 grams	20	20	20			Glass bottle Pomade jar	Refrigerate Refrigerate
Alkalinity	100	75	100	75	75							Pyrex or Polystyrene Pomade jar	Refrigerate See Text
Aluminum	400	400					100					Acid washed Plastic bottle	Refrigerate
			25									Acid washed Plastic bottle	HNO ₃ , 20 drops per liter
				50	100							Glass bottle	HNO ₃ , pH < 2
												Glass bottle	HNO ₃ , 20 drops per liter
												Glass bottle	None
												Pomade jar	None
												Plastic bag	Refrigerate

MINIMUM AMOUNT OF SAMPLE REQUIRED

PARAMETER	Surface Water mL	Well Water mL	Precip. mL	Sewage mL	Ind. Waste mL	Sludge mL	Soil & Sediment grams	Veg'n grams	Bio Material grams	Dust Fall mL	Hi-Vol Air	Preferred Sampling Container	Preservation Technique
Aluminum, cont'd									50				
Anionic Detergents	100	100		100	100	100					1 filter strip	Plastic bag Envelope	Freeze None
Antimony	50	50					2	0.2				Glass bottle Pomade jar Plastic bag	None None Refrigerate
Aromatic Hydrocarbons	1000	1000		1000	1000	20 grams	20	20	20			Glass bottle Pomade jar	Refrigerate Refrigerate
Arsenic	50	50									½ full bag	Sampling bag	Keep in dark cool place
												Glass bottle or plastic	None
				25	25	2	0.2					Glass bottle Pomade jar	None None
								0.2				Plastic bag Plastic bag	Refrigerate Freeze
Asbestos	1000	1000							50			Polystyrene Glassine envelope	Refrigerate None
										1 filter strip			

MINIMUM AMOUNT OF SAMPLE REQUIRED

PARAMETER	Surface Water mL	Well Water mL	Precip. mL	Sewage mL	Ind. Waste mL	Sludge mL	Soil & Sediment grams	Veg'n grams	Bio Material grams	Dust Fall mL	Hi-Vol Air	Ambient Air	Preferred Sampling Container	Preservation Technique
Barium	400	400		50	100								Acid washed plastic bottle	HNO ₃ , 20 drops per liter
													Glass bottle	HNO ₃ , 20 drops per liter
						10							Glass bottle	None
							2						Pomade jar	None
								2					Plastic bag	Refrigerate
									2				Plastic bag	Freeze
Beryllium	400	400											Acid washed plastic bottle	HNO ₃ , 20 drops per liter
				50	100								Glass bottle	HNO ₃ , 20 drops per liter
													Glass bottle	None
						10							Pomade jar	Refrigerate
							2						Plastic bag	Refrigerate
								2					Plastic bag	Freeze
BOD ₅	500	500		500	500								Glass bottle	Refrigerate
Boron	50	50		50	50	50							Plastic bottle	None
							2						Plastic bag	None
								20					Plastic bag	Refrigerate

MINIMUM AMOUNT OF SAMPLE REQUIRED

PARAMETER	Surface Water mL	Well Water mL	Precip. mL	Sewage mL	Ind. Waste mL	Sludge mL	Soil & Sediment grams	Veg'n grams	Bio Material grams	Dust Fall mL	Hi-Vol Air	Ambient Air	Preferred Sampling Container	Preservation Technique
Cadmium	400	400											Acid washed plastic bottle	HNO ₃ 20 drops per liter
			25										Acid washed plastic bottle	HNO ₃ pH <2
				50	100								Glass bottle	HNO ₃ 20 drops per liter
						10							Glass bottle	None
							2						Pomade jar	None
Calcium	100	100											Plastic bag	Refrigerate
	400 (ITC only)	400 (ITC only)							30				Plastic bag	Freeze
													Sampling jar	None
										200			Envelope	None
											1 filter strip			
													Glass or Polystyrene	Refrigerate or none
													Acid washed plastic bottle	HNO ₃ 20 drops per liter
													Polystyrene	Refrigerate
													Acid washed plastic bottle	HNO ₃ pH <2
				50	100								Glass bottle	HNO ₃ 20 drops per liter
													Glass bottle	None
													Pomade jar	None
													Plastic bag	Refrigerate
													Sampling jar	None
										200				

MINIMUM AMOUNT OF SAMPLE REQUIRED

PARAMETER	Surface Water mL	Well Water mL	Precip. mL	Sewage mL	Ind. Waste mL	Sludge mL	Soil & Sediment grams	Veg'n grams	Bio Material grams	Dust Fall mL	Hi-Vol Air	Preferred Sampling Container	Preservation Technique
Carbamate Insecticides & Herbicides	800	800			800							Brown glass solvent rinsed	Refrigerate
Carbon Dioxide	Full bottle	Full bottle										Special bottle See Text	See Text
Carbon Dissolved Inorganic	50	50		50	50	50						Glass bottle	Refrigerate
Carbon Low Level Dissolved Inorganic	Full tube											Pyrex tube with polyseal cap	Refrigerate See Text
Carbon Dissolved Organic	50	50		50	50	50						Glass bottle	Refrigerate
Chemical Oxygen Demand	25	25		25	25	25	2					Glass bottle Pomade jar	Refrigerate None
Chloride	50	50	50	50	50	50						Glass bottle Polystyrene Pomade jar	None Refrigerate None
							20	20				Perforated plastic bag	Refrigerate
										100		Sampling jar Envelope	None None
											1 filter strip		

MINIMUM AMOUNT OF SAMPLE REQUIRED

PARAMETER	Surface Water mL	Well Water mL	Precip. mL	Sewage mL	Ind. Waste mL	Sludge mL	Soil & Sediment grams	Veg'n grams	Bio Material grams	Dust Fall mL	Hi-Vol Air	Ambient Air	Preferred Sampling Container	Preservation Technique
Chlorine - Residual	1000	1000		1000									Glass bottle	See Text
Chlorophenol Insecticides	800	800			800								Brown glass, solvent rinsed	Refrigerate
Chlorophyll	1000												Glass bottle See Text	1 ml 10% MgCO ₃ See Text
Chromium Total	400	400	25										Acid washed Plastic bottle	HNO ₃ , 20 drops per liter
				50	100								Acid washed Plastic bottle Glass bottle	HNO ₃ , pH < 2
						10							Glass bottle	HNO ₃ , 20 drops per liter
							2						Pomade jar	None
								2					Plastic bag	Refrigerate
									50				Plastic bag	Freeze
										200			Sampling jar	None
											1 filter strip		Envelope	None
Chromium Hexavalent	500	500			500								Plastic bottle	None

MINIMUM AMOUNT OF SAMPLE REQUIRED

PARAMETER	Surface Water mL	Well Water mL	Precip. mL	Sewage mL	Ind. Waste mL	Sludge mL	Soil & Sediment grams	Veg'n grams	Bio Material grams	Dust Fall mL	Hi-Vol Air	Preferred Sampling Container	Preservation Technique
Cobalt	400	400		50	100							Acid washed Plastic bottle	HNO ₃ , 20 drops per liter
						10	2					Glass bottle	HNO ₃ , 20 drops per liter
												Glass bottle	None
												Pomade jar	None
								2				Plastic bag	Refrigerate
									50			Plastic bag	Freeze
										200		Sampling jar	None
Colour, Apparent	75	75			75							Glass bottle	Refrigerate
Conductivity	75	75		75	75							Glass bottle or polystyrene	None
			75									Polystyrene	None
							50					Pomade jar	None
Copper	400	400										Acid washed Plastic bottle	HNO ₃ , 20 drops per liter
			25									Acid washed Plastic bottle	HNO ₃ , pH < 2
				50	100							Glass bottle	HNO ₃ , 20 drops per liter
						10						Glass bottle	None
												Pomade jar	None
							2	2				Plastic bag	Refrigerate

MINIMUM AMOUNT OF SAMPLE REQUIRED

PARAMETER	Surface Water mL	Well Water mL	Precip. mL	Sewage mL	Ind. Waste mL	Sludge mL	Soil & Sediment grams	Veg'n grams	Bio Material grams	Dust Fall mL	Hi-Vol Air	Preferred Sampling Container	Preservation Technique
Copper, cont'd													
									50	200		Plastic bag Sampling jar Envelope	Freeze None None
Cyanide	500	500		500	500		10 grams	10				Glass or Plastic bottle Glass bottle	NaOH, pH >10 Refrigerate Keep in dark Refrigerate
Dustfall										30 days exposure		Sampling jar	See Text
Fluoridation Rate											30 days exposure	Calcium oxide candle	Keep sealed when not in use
Fluoride	50	50		50	50	2		0.2				Glass bottle or polystyrene Pomade jar Perforated plastic bag Sampling jar Envelope	None Refrigerate Refrigerate None None
								1.0		100			
											2 filter strips		
Glycols	1000	1000		1000	1000							Glass bottle	Refrigerate

MINIMUM AMOUNT OF SAMPLE REQUIRED

PARAMETER	Surface Water mL	Well Water mL	Precip. mL	Sewage mL	Ind. Waste mL	Sludge mL	Soil & Sediment grams	Veg'n grams	Bio Material grams	Dust Fall mL	Hi-Vol	Ambient Air	Preferred Sampling Container	Preservation Technique
Gold	400	400		50	100								Acid washed plastic bottle	HNO ₃ , 20 drops per liter
						10							Glass bottle	HNO ₃ , 20 drops per liter
							2						Glass bottle	None
								2					Pomade jar	None
									50				Plastic bag	Refrigerate
													Plastic bag	Freeze
Hardness	75	75		75	75								Glass bottle	See Text
Hydrocarbon Gases	1000	1000		1000	1000								Glass bottle	Refrigerate
						20 grams	20	20	20				Pomade jar	Refrigerate
												1/2 full bag	Sampling bag	Keep in dark cool place
Iron (Total)	100	100											Glass bottle or polystyrene	None
	400 (ITC only)	400 (ITC only)											Acid washed Plastic bottle	HNO ₃ , 20 drops per liter
			25										Acid washed plastic bottle	HNO ₃ , pH <2
				50	100								Glass bottle	HNO ₃ , 20 drops per liter
						10							Glass bottle	None

MINIMUM AMOUNT OF SAMPLE REQUIRED

PARAMETER	Surface Water mL	Well Water mL	Precip. mL	Sewage mL	Ind. Waste mL	Sludge mL	Soil & Sediment grams	Veg'n grams	Bio Material grams	Dust Fall mL	Hi-Vol Air	Preferred Sampling Container	Preservation Technique
Iron, cont'd							2					Pomade jar	None
								2				Plastic bag	Refrigerate
									50			Plastic bag	Freeze
										200		Sampling jar	None
											1 filter strip	Envelope	None
Lead	400	400	25									Acid washed plastic bottle	HNO ₃ , 20 drops per liter
												Acid washed plastic bottle	HNO ₃ , pH <2
					100							Glass bottle	HNO ₃ , 20 drops per bottle
				50								Glass bottle	None
						10						Pomade jar	None
Lithium							2					Plastic bag	Refrigerate
									50			Plastic bag	Freeze
										200		Sampling jar	None
											1 filter circle	Envelope	None
												Acid washed plastic bottle	HNO ₃ , 20 drops per liter
Lithium	400	400										Glass bottle	HNO ₃ , 20 drops per liter
				400	400								HNO ₃ , 20 drops per liter

MINIMUM AMOUNT OF SAMPLE REQUIRED

PARAMETER	MINIMUM AMOUNT OF SAMPLE REQUIRED										Hi-Vol	Ambient Air	Preferred Sampling Container	Preservation Technique
	Surface Water mL	Well Water mL	Precip. mL	Sewage mL	Ind. Waste mL	Sludge mL	Soil & Sediment grams	Veg'n grams	Bio Material grams	Dust Fall mL				
Lithium, cont'd						10							Glass bottle	None
						2							Pomade jar	None
							2						Plastic bag	Refrigerate
								50					Plastic bag	Freeze
										200			Sampling jar	None
Magnesium	100	100											Glass bottle or polystyrene	Refrigerate or none
	400 (ITC only)	400 (ITC only)											Acid washed plastic bottle	HNO ₃ , 20 drops per liter
			100										Polystyrene	None
			25 (ITC only)										Acid washed plastic bottle	HNO ₃ pH <2
				50	100								Glass bottle	HNO ₃ , 20 drops per liter
						10							Glass bottle	None
							2						Pomade jar	None
								2					Plastic bag	Refrigerate
									50				Plastic bag	Freeze
										200			Sampling jar	None
										1 filter strip		Envelope	None	

MINIMUM AMOUNT OF SAMPLE REQUIRED

PARAMETER	Surface Water mL	Well Water mL	Precip. mL	Sewage mL	Ind. Waste mL	Sludge mL	Soil & Sediment grams	Vegh grams	Bio Material grams	Dust Fall mL	Hi-Vol Air	Ambient Air	Preferred Sampling Container	Preservation Technique
Manganese	75	75											Glass bottle or polystyrene	None
	400	400											Acid washed plastic bottle	HNO ₃ , 20 drops per liter
	(ITC only)	(ITC only)	25										Acid washed plastic bottle	HNO ₃ , pH < 2
				50	100								Glass bottle	HNO ₃ , 20 drops per liter
						10							Glass bottle	None
Matrix Elements in Vegetation							2						Pomade jar	None
								2					Plastic bag	Refrigerate
									50				Plastic bag	Freeze
										200			Sampling jar	None
											1 filter strip		Envelope	None
Mercury	176	176	176										Perforated plastic bag	Refrigerate
								2					Use special bottle	HNO ₃ + K ₂ Cr ₂ O ₇
													See Text	
				100	100								Glass bottle	HNO ₃ + K ₂ Cr ₂ O ₇
						500							Glass bottle	HNO ₃
							10						Pomade jar	None
								3 (dried)					Plastic bag	Freeze

MINIMUM AMOUNT OF SAMPLE REQUIRED

PARAMETER	Surface Water mL	Well Water mL	Precip. mL	Sewage mL	Ind. Waste mL	Sludge mL	Soil & Sediment grams	Veg'n grams	Bio Material grams	Dust Fall mL	Hi-Vol Air	Ambient Air	Preferred Sampling Container	Preservation Technique
Microbiological Indicators of Fecal Pollution														
E. coli	150	150		150									Presterilized glass with Thiosulphate	Pack in ice
Fecal Coliforms	150	150		150									Presterilized glass with Thiosulphate	Pack in ice
Fecal Streptococci	150	150		150									Presterilized glass with Thiosulphate	Pack in ice
Presence/Absence	150	150		150									Presterilized glass with Thiosulphate	Pack in ice
P. aeruginosa	150	150		150									Presterilized glass with Thiosulphate	Pack in ice
Salmonella	1000	1000		1000									Presterilized glass with Thiosulphate	Pack in ice
Total Coliforms	150	150		150									Presterilized glass with Thiosulphate	Pack in ice
Microbiological Indicators of Specific Pollution														
K. pneumoniae	150	150		150									Presterilized glass with Thiosulphate	Pack in ice
Nitrogen cycle Bacteria	150	150		150									Presterilized glass with Thiosulphate	Pack in ice
Phenol degrading Bacteria	150	150		150									Presterilized glass with Thiosulphate	Pack in ice
Sulphate Reducing Bacteria	150	150		150									Presterilized glass Consult staff	Pack in ice
Sulphur Oxidizing Bacteria	150	150		150									Presterilized glass	Pack in ice

MINIMUM AMOUNT OF SAMPLE REQUIRED

[illegible]

MINIMUM AMOUNT OF SAMPLE REQUIRED

PARAMETER	Surface Water mL	Well Water mL	Precip. mL	Sewage mL	Ind. Waste mL	Sludge mL	Soil & Sediment grams	Veg'n grams	Bio Material grams	Dust Fall mL	Hi-Vol Air	Preferred Sampling Container	Preservation Technique
Nickel	400	400										Acid washed plastic bottle	HNO ₃ , 20 drops per liter
			25									Acid washed Plastic bottle	HNO ₃ , pH <2
				50	100							Glass bottle	HNO ₃ , 20 drops per liter
						10						Glass bottle	None
							2					Pomade jar	None
								2				Plastic bag	Refrigerate
									50			Plastic bag	Freeze
										200		Sampling jar	None
											1 filter strip	Envelope	None
Nitrogen Ammonia and Ammonium	75	75		75	75	75						Glass bottle or polystyrene	Refrigerate or freeze
			75									Polystyrene	Refrigerate
							2					Pomade jar	Refrigerate
												Envelope	None
											1 filter strip		
Nitrogen Nitrate	75	75		75	75	75						Glass bottle or polystyrene	Refrigerate or freeze
			75									Polystyrene	Refrigerate
							2					Pomade jar	Refrigerate
												Envelope	None
											1 filter strip		

MINIMUM AMOUNT OF SAMPLE REQUIRED

PARAMETER	Surface Water mL	Well Water mL	Precip. mL	Sewage mL	Ind. Waste mL	Sludge mL	Soil & Sediment grams	Veg'n grams	Bio Material grams	Dust Fall mL	Hi-Vol Air	Ambient Air	Preferred Sampling Container	Preservation Technique
Nitrogen Nitrite	75	75	75	75	75	75							Glass bottle or polystyrene Pomade jar	Refrigerate or freeze Refrigerate
Nitrogen Total Kjeldahl	75	75	75	75	75	75							Glass bottle or polystyrene Pomade jar Plastic jar	Refrigerate or freeze Refrigerate Refrigerate
Nitrotoluenes	1000	1000		1000	1000								Glass bottle	Refrigerate
Organic Solvents	1000	1000		1000	1000	20 grams	20	20	20			1/2 full bag	Glass bottle Pomade jar Sampling bag	Refrigerate Refrigerate Keep in dark cool place
Organo-Chlorine Pesticides and PCB's	800	800			800								Brown glass solvent rinsed Special jar Florisil Tube	Refrigerate Refrigerate Refrigerate
Organo-Halides Volatile	1000	1000		1000	1000							10 cubic metres	Glass bottle	Refrigerate

MINIMUM AMOUNT OF SAMPLE REQUIRED

PARAMETER	Surface Water mL	Well Water mL	Precip. mL	Sewage mL	Ind. Waste mL	Sludge mL	Soil & Sediment grams	Vegh grams	Bio Material grams	Dust Fall mL	Hi-Vol	Ambient Air	Preferred Sampling Container	Preservation Technique
Organo Phosphorus Insecticides	800	800			800			10	10				Brown glass solvent rinsed Special jar	Refrigerate
Organo-Sulphur Compounds	1000	1000		1000	1000	20 grams	20	20	20			½ full bag	Glass bottle Pomade jar Sampling bag	Refrigerate Refrigerate Keep in dark cool place
Oxygen Dissolved	Full bottle	Full bottle											Glass bottle See Text	Refrigerate Keep in dark
Particle Size Distribution					25								Pomade jar	None
Petroleum Hydrocarbons	1000	1000		1000	1000	30 grams	30	30					Glass bottle See Text Pomade jar	Refrigerate Refrigerate
pH	100	50	100	50	50	50							Hard glass or polystyrene Polystyrene Pomade jar	See text See text Refrigerate

MINIMUM AMOUNT OF SAMPLE REQUIRED

PARAMETER	Surface Water mL	Well Water mL	Precip. mL	Sewage mL	Ind. Waste mL	Sludge mL	Soil & Sediment grams	Veg'n grams	Bio Material grams	Dust Fall mL	Hi-Vol	Ambient Air	Preferred Sampling Container	Preservation Technique
Nitrogen Nitrite	75	75	75	75	75	75							Glass bottle or polystyrene Pomade jar	Refrigerate or freeze Refrigerate
Nitrogen Total Kjeldahl	75	75	75	75	75	75							Glass bottle or polystyrene Pomade jar Plastic jar	Refrigerate or freeze Refrigerate Refrigerate
Nitrotoluenes	1000	1000	1000	1000	1000			0.5					Glass bottle	Refrigerate
Organic Solvents	1000	1000	1000	1000	1000	20 grams	20	20	20			½ full bag	Glass bottle Pomade jar Sampling bag	Refrigerate Refrigerate Keep in dark cool place
Organo-Chlorine Pesticides and PCB's	800	800			800								Brown glass solvent rinsed Special jar Florisil Tube	Refrigerate Refrigerate Refrigerate
Organo-Halides Volatile	1000	1000	1000	1000	1000							10 cubic metres	Glass bottle	Refrigerate

MINIMUM AMOUNT OF SAMPLE REQUIRED

PARAMETER	MINIMUM AMOUNT OF SAMPLE REQUIRED											Preservation Technique		
	Surface Water mL	Well Water mL	Precip. mL	Sewage mL	Ind. Waste mL	Sludge mL	Soil & Sediment grams	Veg'n grams	Bio Material grams	Dust Fall mL	Hi-Vol		Ambient Air	Preferred Sampling Container
Organo-Phosphorus Insecticides	800	800			800								Brown glass solvent rinsed Special jar	Refrigerate
							10	10						Refrigerate
Organo-Sulphur Compounds	1000	1000		1000	1000	20 grams	20	20	20				Glass bottle Pomade jar	Refrigerate
												½ full bag	Sampling bag	Refrigerate
Oxygen Dissolved														Keep in dark cool place
	Full bottle	Full bottle											Glass bottle See Text	Keep in dark
Particle Size Distribution							25						Pomade jar	None
Petroleum Hydrocarbons	1000	1000		1000	1000	30 grams	30						Glass bottle See Text Pomade jar	Refrigerate
														Refrigerate
pH	100	50		50	50	50							Hard glass or polystyrene Polystyrene Pomade jar	See text
			100											See text
							10						Pomade jar	Refrigerate

MINIMUM AMOUNT OF SAMPLE REQUIRED

PARAMETER	Surface Water mL	Well Water mL	Precip. mL	Sewage mL	Ind. Waste mL	Sludge mL	Soil & Sediment grams	Vegh grams	Bio Material grams	Dust Fall mL	Hi-Vol Air	Ambient Air	Preferred Sampling Container	Preservation Technique
Phosphorus Total, by Dissolution	See Phosphorus in Soil													
Phthalic Acid esters	1000	1000		1000	1000								Glass bottle	Refrigerate
Polynuclear Aromatic Hydrocarbons (PAH)	1000	1000		1000	1000	20 grams	20	20	20				Glass bottle Pomade jar Envelope	Refrigerate Refrigerate None
Potassium	50	50	50	50	50						1 filter strip	1/2 full bag	Sampling bag	Keep in dark cool place
			50										Glass bottle or polystyrene	None
													Polystyrene	Refrigerate
													Pomade jar	None
													Plastic bag	Refrigerate
													Plastic bag	Freeze
Radionuclides	8 + mix	8 + mix											Special vial	Consult staff
Selenium	50	50		25	25	2	0.2						Glass bottle	None
													Pomade jar	None
													Plastic bag	Refrigerate
													Plastic bag	Freeze

MINIMUM AMOUNT OF SAMPLE REQUIRED

PARAMETER	Surface Water mL	Well Water mL	Precip. mL	Sewage mL	Ind. Waste mL	Sludge mL	Soil & Sediment grams	Vegh grams	Bio Material grams	Dust Fall mL	Hi-Vol Air	Preferred Sampling Container	Preservation Technique
Silicon and Silicates - Reactive	50	50		50								Polyethylene bottle	None
Silver	400	400										Acid washed plastic bottle	HNO ₃ , 20 drops per liter
				50	100							Glass bottle	HNO ₃ , 20 drops per liter
						10	2					Glass bottle	None
												Pomade jar	None
Sodium	50	50	50	50	50							Pyrex bottle or polystyrene	Refrigerate or none
						10						Polystyrene	Refrigerate
												Pyrex bottle	None
												Pomade jar	None
												Plastic bag	Refrigerate
										200		Sampling jar	None
Solids Dissolved	75	75		75	75							Glass bottle	Refrigerate
Solids Ignited	600	600		600	600	50						Glass bottle	Refrigerate
Solids Suspended	600	600		600	600							Glass bottle	Refrigerate

MINIMUM AMOUNT OF SAMPLE REQUIRED

PARAMETER	Surface Water mL	Well Water mL	Precip. mL	Sewage mL	Ind. Waste mL	Sludge mL	Soil & Sediment grams	Veg'h grams	Bio Material grams	Dust Fall mL	Hi-Vol	Ambient Air	Preferred Sampling Container	Preservation Technique
Solids Total	75	75		75	75	50							Glass bottle	Refrigerate
Strontium	400	400		50	100								Acid washed plastic bottle	HNO ₃ , 20 drops per liter
													Glass bottle	HNO ₃ , 20 drops per liter
						10							Glass bottle	None
							2						Pomade jar	None
								2					Plastic bag	Refrigerate
									50				Plastic bag	Freeze
Sulphate	50	50		50	50								Glass bottle or polystyrene	None
			50										Polystyrene	Refrigerate
							50						Pomade jar	None
													Sampling jar	None
													Envelope	None
										5	1 filter strip			
Sulphation Rate												30 days exposure	Lead dioxide coated filter	Keep sealed before and after use
Sulphide	1000	25		1000	1000	50							Glass bottle	Refrigerate
							2						Pomade jar	None
Suspended Air particulate												1 day exposure	Envelope	Keep dry

MINIMUM AMOUNT OF SAMPLE REQUIRED

PARAMETER	Surface Water mL	Well Water mL	Precip. mL	Sewage mL	Ind. Waste mL	Sludge mL	Soil & Sediment grams	Veg'n grams	Bio Material grams	Dust Fall mL	Hi-Vol Air	Ambient Air	Preferred Sampling Container	Preservation Technique
Tannins and Lignins	200				200								Glass bottle	Refrigerate
Thallium	400	400	25		50	100							Acid washed plastic bottle	HNO ₃ , 20 drops per liter
													Acid washed plastic bottle	HNO ₃ pH <2
													Glass bottle	HNO ₃ , 20 drops per liter
													Glass bottle	None
													Pomade jar	None
													Plastic bag	Refrigerate
													Plastic bag	Freeze
Tin	400	400											Acid washed plastic bottle	HNO ₃ , 20 drops per liter
													Glass bottle	HNO ₃ , 20 drops per liter
													Glass bottle	None
													Pomade jar	None
													Plastic bag	Refrigerate
Titanium	400	400	25										Acid Washed plastic bottle	HNO ₃ , 20 drops per liter
													Acid washed plastic bottle	pH <2

MINIMUM AMOUNT OF SAMPLE REQUIRED

PARAMETER	Surface Water mL	Well Water mL	Precip. mL	Sewage mL	Ind. Waste mL	Sludge mL	Soil & Sediment grams	Veg'h grams	Bio Material grams	Dust Fall mL	Hi-Vol Air	Preferred Sampling Container	Preservation Technique
Titanium, cont'd				50	100							Glass bottle	HNO ₃ , 20 drops per liter
						10						Glass bottle	None
							2					Pomade jar	None
								2	50			Plastic bag	Refrigerate
												Plastic bag	Freeze
Tracer Dyes	100				100							Glass bottle	Refrigerate Keep in dark
Triazine Herbicides	800	800			800							Brown glass solvent rinsed Special jar	Refrigerate
							10	10				Glass bottle or polystyrene	Refrigerate
Turbidity	50	50		50	50								Keep in dark
Uranium	400	400			400							Acid washed plastic bottle	HNO ₃ , 20 drops per liter
												Glass bottle	HNO ₃ , 20 drops per liter
Vanadium	400	400										Acid washed plastic bottle	HNO ₃ , 20 drops per liter
												Acid washed plastic bottle	HNO ₃ , pH <2

MINIMUM AMOUNT OF SAMPLE REQUIRED

PARAMETER	Surface Water mL	Well Water mL	Precip. mL	Sewage mL	Ind. Waste mL	Sludge mL	Soil & Sediment grams	Veg'h grams	Bio Material grams	Dust Fall mL	Hi-Vol Air	Preferred Sampling Container	Preservation Technique
Vanadium, cont'd				50	100								
						10	2					Glass bottle	HNO ₃ 20 drops per liter
												Glass bottle	None
												Pomade jar	None
												Plastic bag	Refrigerate
												Plastic bag	Freeze
												Sampling jar	None
												Envelope	None
										200	1 filter strip		
Vinyl Chloride	1000	1000		1000	1000							Glass bottle	Refrigerate
						20 grams	20	20	20			Pomade jar	Refrigerate
												Sampling bag	Keep cool Keep in dark
											1/2 full bag		
Volatile Acids				25		25						Glass bottle	Refrigerate
Zinc	400	400										Acid washed plastic bottle	HNO ₃ 20 drops per liter
			25									Acid washed plastic bottle	HNO ₃ pH < 2

MINIMUM AMOUNT OF SAMPLE REQUIRED

PARAMETER	Surface Water mL	Well Water mL	Precip. mL	Sewage mL	Ind. Waste mL	Sludge mL	Soil & Sediment grams	Veg'n grams	Bio Material grams	Dust Fall mL	Hi-Vol Air	Ambient Air	Preferred Sampling Container	Preservation Technique
Zinc, cont'd				50	100								Glass bottle	HNO ₃ , 20 drops per liter
						10							Glass bottle	None
							2						Pomade jar	None
								2					Plastic bag	Refrigerate
									50				Plastic bag	Freeze
										200			Sampling jar	None
											1 filter strip		Envelope	None



TABLE 3**SPECIAL PACKS****NOTICE:**

Central Stores will require forty-eight (48) hours notice for any special pack orders as these packs are not stocked on a regular basis.

PACK NUMBER	DESCRIPTION
1	1 x liter, glass, styrofoam, single cradle
3	6 x 1 liter, glass
3 P.C.B.	6 x 1 liter, glass
3 S	6 x 1 liter, glass, special caps
3 L	6 x 1 liter, glass, special Lim & Tox distilled
4	6 x 32 oz. glass, wide mouth
5	12 x 16 oz. glass, wide mouth
5 P.C.B.	12 x 16 oz. glass, wide mouth
6 T	2 x 6 oz. glass, Thio, Red Label
6 P	2 x 6 oz. glass, Phenol Green Label
7 C	4 x 6 oz. glass
7 T	4 x 6 oz. glass, Thio, Red Label
7 P	4 x 6 oz. glass, Phenol Green Label
8 C	72 x 6 oz. glass
8 T	72 x 6 oz. glass, Thio, Red Label
9	6 x 32 oz. plastic, wide mouth
10	6 x 32 oz. plastic, wide mouth, acid wash
11	6 x 16 oz. plastic, wide mouth
11 A	6 x 16 oz. plastic, acid wash
12	12 x 8 oz. plastic, polystyrene
13	9 x 16 oz. plastic, polystyrene
15	4 x 6 oz. Ice pack
16	4 x 32 oz. asbestos
Health Pack	10 bottles

MANUAL REVISION CARD

Manual User

Last Name

Initial

Address to send future revisions or additions:

Organization

Street

City

Province

Postal Code

Date

Signature

Cut here

Ministry of the Environment
Laboratory Services Branch
Water Quality Section
Box 213
Rexdale, Ontario.
M9W 5L1